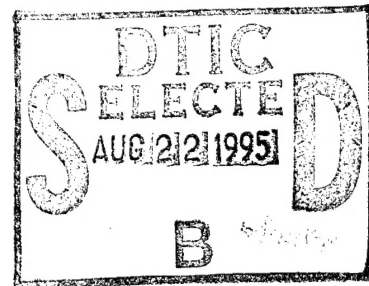


# **EFFECT OF FUEL COMPOSITION AND PRESTRESSING ON LUBRICITY**

**INTERIM REPORT  
TFLRF No. 307**



By

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13. ABSTRACT (Maximum 200 words)  Fuel lubricity--or the ability of the fuel to prevent wear during sliding--is sensitive to chemical composition. At present, increasingly severe fuel specifications are being implemented to minimize exhaust emissions. The refinery processes needed to achieve these goals are inadvertently removing many of the surface-active components necessary for wear resistance. To compound this effect, engine operating conditions are becoming more severe; fuel injection pressures are increasing dramatically, while uncooled engines that use the fuel as a heat sink are also being investigated. The present study investigates the relationship between previously validated laboratory-scale wear tests and various fuels' parameters. In general, fuel lubricity is found to be adversely affected by decreasing sulfur and aromatics content, and appears to be most closely related to diaromatics. Laboratory wear tests and full-scale equipment tests were performed to define the effects of operating temperature and thermal prestressing of the fuel. The effects of temperature on wear appear to be fuel composition sensitive. Indeed, the corrosion inhibitor additives tested had little effect at high temperatures. As a result, it is likely that conventional laboratory-scale wear tests performed at room temperature may not fully reflect real-world operating conditions.				
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## EXECUTIVE SUMMARY

**Problems:** The composition of military and commercial fuels intended for use in compression ignition vehicles is changing. Diesel fuels are being more severely refined to minimize exhaust emissions, while use of nontraditional fuels (such as Jet A-1, JP-8, and biofuel) is also becoming more common. The effects of these changes on equipment durability have not been fully defined. The problem is compounded by increasing injection pressures and temperatures in future military vehicles.

**Objective:** The objective of this project was to define the effect of fuel composition and prestressing on lubricity.

**Importance of Project:** The fuel injection system is central to the reliable operation of compression ignition engines. An improved understanding of the fuel characteristics responsible for lubricity, as well as the operating conditions which adversely affect lubricity, will assure acceptable durability. Recirculated fuel is commonly used to cool the injection system and has the potential for deposit formation under severe operating conditions. The effect of these deposits or even removal of the compounds responsible for deposit formation is unknown.

**Technical Approach:** A large database exists for the lubricity of fuels tested using the U.S. Army Scuffing Load Wear Test (SLWT) procedure (which shows good correlation with full-scale equipment). The database includes samples provided by the U.S. military, commercial sponsors, and the International Standards Organization (ISO). This report correlates the measured lubricity with any available physical and chemical characteristics. A number of military vehicle field failures is also discussed. Testing was performed to define the effects of high temperature operation and thermally prestressing the fuel to remove compounds responsible for deposit formation on lubricity.

**Accomplishments:** A strong correlation was observed between fuel lubricity and refinery severity, as measured by sulfur and aromatic content. A number of other parameters also showed a slight correlation with lubricity. Thermal prestressing using a single tube heat exchanger improved the lubricity of severely refined fuels. However, little correlation was observed between laboratory-scale wear tests performed at room temperature and full-scale injector tests performed at high temperature.

**Military Impact:** The results of this study indicate that a small number of commercially available diesel fuels have low lubricity, which may promote severe injection system wear. A significantly larger number of kerosene fuels have low lubricity. Blending these fuels with a small volume of better lubricity fuel significantly improves overall wear resistance, and occurs naturally in most vehicle fuel tanks. Larger users that obtain fuel from a single source, such as the Department of Defense (DOD), are especially susceptible to the effects of isolated, low lubricity fuels. Boundary lubrication of fuel-lubricated components is sensitive to operating temperature. A revised laboratory-scale wear test procedure may be required to predict wear in high temperature applications.



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## I. INTRODUCTION

The United States and Europe are mandating increasingly severe diesel fuel specifications, particularly with respect to sulfur content, and in some areas, aromatics content. This trend is directed towards reducing vehicle exhaust emissions and is generally beneficial to fuel quality, ignition ratings, and stability. However, laboratory studies, as well as recent field experience in Sweden and the United States, indicate a possible reduction in the ability of fuels to lubricate sliding components within the fuel injection system. These factors, combined with the trend toward increasing injection pressures, are likely to result in reduced durability and failure of the equipment to meet long-term emissions compliance.

In addition, future military engines are expected to have reduced heat loss and associated higher operating temperatures. These changes may eliminate the vulnerable cooling system and improve efficiency. The engines being developed typically rely on excess fuel returned to the fuel cell to cool the injection system and cylinder head, resulting in increased deposit formation in the high temperature fuel system. Thermally prestressing the fuel prior to entering the engine may remove the deposit-forming compounds. However, the effects of thermally prestressing the fuel on its ability to prevent wear have not been defined. Indeed, until recently, no accurate test procedure was available to measure fuel lubricity.

The U.S. Army TARDEC Fuels and Lubricants Research Facility (TFLRF) located at Southwest Research Institute (SwRI) developed an accelerated wear test that predicts the effects of fuel lubricity on injection system durability. Described in previous reports (1, 2)\*, this test now has been widely used by fuel, additive, and equipment manufacturers. Several thousand fuel samples have been evaluated to date, and collectively, they form one of the largest databases on fuel lubricity currently in existence. This range of data permits a good overview of the commercially available fuels and confirms a general decrease in fuel lubricity due to increased refining severity. The results indicate that no high-sulfur fuel ( $>0.15$  mass%) had poor lubricity, while a number of low-sulfur fuels ( $<0.05$  mass%) did produce unacceptable wear. The lubricity of many highly refined fuels is probably being restored using relatively high concentrations of additive.

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\* Underscored numbers in parentheses refer to the list of references at the end of this report.

The laboratory-scale wear tests were also used to define the effects of thermal prestressing on the lubricity of several fuels, with a wide range of chemical compositions. In addition, full-scale equipment tests were performed to examine the effects of various fuels' parameters and operating temperature on seizure of unit injection systems.

## **II. OBJECTIVE**

The objective of the present study is to define the effects of various fuel characteristics on lubricity. The parameters to be evaluated include chemical and physical composition, as well as the effects of thermal prestressing and high temperature operation.

## **III. BACKGROUND**

In the United States, visible smoke from heavy-duty diesel engines has been subject to regulation by the Environmental Protection Agency (EPA) since 1970. Increasingly severe regulations for hydrocarbon (HC), oxides of nitrogen ( $\text{NO}_x$ ), and particulate matter (PM) emissions were implemented throughout the 1980s and into the 1990s. Initially, these requirements were achieved through improvements in the combustion system (comprised of the combustion chamber, ignition equipment, ignition timing, etc.).

However, the increasingly stringent particulate requirements implemented in the 1990s require modifications to the composition of the fuel as well as the engine hardware to obtain further reductions of emissions. The United States and Europe are mandating increasingly severe diesel fuel specifications, particularly with respect to sulfur content and in some areas, aromatics content. The trend is generally beneficial to fuel quality, ignition ratings, and stability. However, laboratory studies, as well as recent field experience in Sweden and the United States, indicate a possible reduction in the ability of fuels to lubricate sliding components within the fuel injection system.(3-5)

Diesel fuel specifications have intentionally remained broad to permit the maximum availability of the fuel and lowest price possible.(6) Historically, many developed nations have allowed a fairly high sulfur content in the range of 0.5 mass percent (mass%), with no specification on aromatics content.(7) The major industrialized nations are currently tightening diesel fuel quality specifications, with elimination of sulfur being the primary concern. Reduction in sulfur content will minimize formation of sulfuric acid in the atmosphere and will facilitate future development of platinum-catalyzed particulate traps.(6) Data generated in a cooperative study sponsored by the Coordinating Research Council (CRC) as part of their Vehicle Emissions Program (VE 1) confirmed the primary importance of sulfur in emissions.(8, 9) However, the VE 1 study indicated that exhaust hydrocarbons, carbon monoxide, oxides of nitrogen, and particulate matter were also marginally reduced by decreasing aromatics content. Later studies have indicated that cetane number may be more directly related to emissions than to aromatics concentration alone.(10, 11)

A selection of the relevant fuel specifications from around the world is summarized in TABLE 1. In 1991 and 1992, Sweden defined several new classes of fuel that regulate a number of fuel characteristics, including sulfur and aromatics content. Different tax classes were designed to provide economic incentives to use the clean burn Class I and II fuels rather than the higher sulfur Class III fuel. Similarly, in the United States, federal regulations implemented by the EPA limited the maximum fuel sulfur content to 0.05 mass% from its previous level of 0.5 mass%, effective October 1993. Additionally, the aromatics were limited by either a 40 cetane index minimum or a 35 vol% aromatics limit. Based on the VE 1 study, the California Air Resources Board (CARB) mandated an additional requirement of 10 vol% aromatics minimum in diesel fuel sold in California. Alternatively, refiners could certify diesel fuel formulations with equivalent or lower emissions when compared to a CARB-specified, 10 vol% aromatics reference fuel. Lowering the aromatics content of diesel fuels from the previous levels of well over 30 vol% to the 5 or 10 vol% mandated by the newer regulations requires severe hydrotreatment (i.e., reaction of intermediate petroleum products with hydrogen) for many refineries. As a result, some California fuel producers are minimizing the level of refinery processing by developing alternate fuels that will demonstrate equivalent emissions and will have slightly higher aromatics content.(12)

**TABLE 1. Fuel Classes**

Fuel Class	Introduced	Maximum Sulfur, ppm	Maximum Aromatics, vol%	Minimum Cetane Number	90% Point, °C	Mean SLC*, grams
Sweden/Class I	1991	10	5	50	285 (95%)	1,200**†
Sweden/Class II	1991	50	5	47	295 (95%)	1,400**†
Sweden/Class III	1991	3,000	--	46	340	--
Europe	1994	2,000	--	48	--	3,800†
Europe/CEN	1996	500	--	49	370	N/A‡
Japan	Pre-1997	2,000	--	45	350	N/A
	Post-1997	500	--	45	350	N/A
USA/VV-F-800	Pre-1993	5,000	--	40	338	3,866
USA/EPA	Post-1993	500	35§	40	338	3,086
USA/CARB	Post-1993	500	10◆	48	320	3,081

\* SLC = Scuffing Load Capacity

\*\* Later Swedish fuels that contained lubricity additives had an SLC in excess of 3,000 grams.

† Represents data obtained from a small number of samples.

‡ N/A = Not yet available

§ Aromatics limited to 35 vol% or a minimum cetane index of 40.

◆ Fuel intended for use in California must have an aromatics content below 10 vol% unless shown to produce emissions below that of a CARB-specified referee fuel.

The U.S. Army has a number of additional requirements that are unique to its vehicles. Low heat rejection engines--also referred to as adiabatic engines--and other high temperature, high output diesel engines are being considered by the U.S. Army as powerplants for future ground vehicles. An insulated low heat rejection engine configuration affords many important advantages of military interest. These advantages include compact engine size, lower engine weight/power output, less smoke, and improved specific fuel consumption.(13-18) However, all current engine designs require some degree of cooling. Many use the incoming fuel as a heat sink to reduce the temperature of the critical cylinder head area. Excess unburned fuel is returned, carrying some heat to the fuel cell. However, thermal oxidation and even pyrolysis of the fuel may produce deposits within the injection system, depending on operating temperature and fuel quality. A number of solutions have previously been evaluated, including optimizing the metallurgy present.(1) Ideally, the fuel components responsible for deposit formation would be removed prior to entering the diesel fuel injection system.



A mechanism to thermally prestress the fuel to remove the least stable components prior to entering the fuel injection system is described in References 1 and 2. A single tube heat exchanger (STHE) was developed and is capable of operating at temperatures of up to 500°C. However, the boundary lubricating qualities of the fuel may be affected by removal of these more reactive compounds. Indeed, high flash temperatures occurring during wear may produce polymeric surface deposits (i.e., hydroperoxides and carboxylic acids which are very similar to those formed during the thermal oxidation process). Artificially removing these compounds may either increase or reduce the overall wear rate, depending on the predominant lubrication mechanisms present.

A high concentration of vehicles and equipment within DOD utilize fuel-lubricated rotary fuel injection pumps that are particularly sensitive to fuel lubricity. A previous U.S. Army-sponsored study on fuel lubricity indicated that wear in the injection system (3, 4, 19-22) was not accurately measured by any existing laboratory-scale wear test for fuels. As a result, a test procedure that uses the Ball-on-Cylinder Lubricity Evaluator (BOCLE) was developed and is commonly referred to as the U.S. Army Scuffing Load Wear Test (SLWT).(23) Previously published results based on the Army SLWT using a small number of fuels indicated a directional correlation between fuel lubricity and refining severity.(5) In the present study, the Army SLWT and other tests are used to 1) define the effects of fuel prestressing and composition on lubricity and 2) define the effect of various parameters on failure in unit fuel injection systems, with particular respect to the effects of temperature.

## **IV. EXPERIMENTAL**

### **A. Laboratory-Scale Test Equipment**

The majority of laboratory-scale tests were performed using the BOCLE according to the Army SLWT procedure. The BOCLE was also used to perform the procedure defined in ASTM D 5001 (24), which has been widely used for a number of years. The principal test conditions of both procedures are summarized in TABLE 2.

---

**TABLE 2. Operating Conditions for Laboratory Wear Tests**

Test Parameter	BOCLE (Army SLWT)	BOCLE (ASTM D 5001)	Cameron- Plint
Applied Load, kg	1 to 5 (variable)	0.5	1 to 25
Speed	525 rpm	240 rpm	50 Hz
Break-in, sec/kg	30/0.5	None	None
Duration, min	1.0	30	10
Atmosphere	Controlled air	Controlled air	Uncontrolled
Humidity, %Rh	50	10	Uncontrolled
Temperature, °C	25	25	40
Pass/Fail	3 kg	0.65 mm	N/A*

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\* N/A = Not applicable

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The ASTM D 5001 test consists of a 12.7-mm diameter steel ball in contact with a 49.5-mm diameter rotating cylinder. Sliding contact occurs over a 30-minute period, and the average diameter of the elliptical wear scar is taken as a measure of fuel lubricity. To ensure accurate control of the surface oxidative wear process, the test specimens and fuel are enclosed in a controlled environment of variable humidity and temperature. A complete description of the ASTM D 5001 test procedure and its application may be obtained in References 21 and 23.

The Army SLWT determines the minimum applied load required for a step transition to adhesive scuffing. (A complete description of the test and its development may be obtained in References 23 and 25.) During testing, the fluid is placed in a humidity-controlled reservoir. A nonrotating steel ball is held in a vertically mounted chuck and forced against an axially mounted polished steel ring. A sequence of one-minute tests is performed, and the applied load is systematically changed until a disproportionate change in friction and wear is observed. The fuel is not renewed between load increments during a normal test sequence. The test cylinder is rotated at a fixed speed while being partially immersed in the fluid reservoir. The minimum applied load required to produce a transition to severe friction and wear is a measure of the fluid-lubricating properties and is inversely related to lubricity. An Army SLWT result of 3,000 grams is believed to approximate the transition between a poor and an acceptable lubricity fuel (e.g., an Army SLWT result in excess of 3,000 grams should provide acceptable equipment durability). A more

complete discussion of the significance of the laboratory wear test results is provided in Appendix A.

A number of wear tests were performed using the Cameron-Plint high-frequency reciprocating wear test. In this test, the upper specimen slides on a stationary lower flat with a pure sinusoidal motion. The scotch yoke mechanism that provides this motion is driven by a variable speed motor. This machine provides the relatively low contact stresses required for effective wear testing of fuels. In addition, the oscillating contact is analogous to that found in many areas of the injection system. The principal test conditions are summarized in TABLE 2. The test specimens consist of a 6.35-mm diameter ball sliding on a polished test flat. Wear is represented by the diameter of the scar formed on the ball and is measured using optical microscopy. If the scar is not circular, an average diameter is calculated from measurements taken along the major and minor axes of the ellipse formed.

## **B. Full-Scale Test Equipment**

Full-scale equipment tests were performed using a Detroit Diesel N70 unit injector motored on a test stand. The objective of these tests was to simulate the effects of fuel lubricity and viscosity on unit injection system failures in a real application. A schematic diagram of the unit injector is provided in Fig. B-1. The relatively simple operating mechanism of the unit injection system minimizes the number of wear contacts present. This, combined with the extremely good durability of the design, necessitates an accelerated test procedure.

A common failure mechanism for unit injection systems in many full-scale applications is via catastrophic seizure between the plunger and barrel. Such seizures may occur after an extended time period and require a relatively large database to obtain a good statistical average. Factors contributing to seizure include misalignment between components, distortion of the injector body due to fuel pressure, and swelling or bending of the plunger during the injection stroke. In the present study, these conditions were simulated by artificially side loading the injector plunger by way of the injector gear, shown in Appendix B. During normal operation of the injector, the plunger slides through the injector gear to form a loose slip fit with little side loading. The

purpose of the gear and rack mechanism is to control the fuel delivery rate by rotating the plunger. The remainder of the plunger slides within the injector body and forms a very close seal to minimize fuel leakage.

To allow application of the side load, a 4-mm diameter hole was machined in the side of the injector body in line with the gear mechanism. The hole was formed using an electronic discharge machine to minimize distortion of the injector body. Very low fuel pressure is present at this point, and significant fuel leakage does not occur. A 4-mm diameter metal rod connected to a dead weight loading system was inserted through the hole. The injector rack was removed to allow the end of the rod to come into direct contact with the gear. The loading mechanism was designed to allow a variable side load of between 0 and 20 kg to be applied to the gear and plunger. This load range was sufficient to produce seizure with each of the fluids evaluated. TABLE 3 summarizes the principal test parameters for the unit injector.

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**TABLE 3. Principal Test Conditions for Detroit Diesel Unit Injector**

<u>Parameter</u>	<u>Value</u>
Speed, rpm	2,000
Temperatures, °C	
Fuel inlet	38
Heating block	150, 200, 290
Side load, kg	0 to 20
Throttle	Wide open

---

During testing, an electric motor-driven cam operates the injector plunger. The side loading on the gear and plunger is increased incrementally until seizure is observed. Post-failure disassembly of the unit injectors indicates that seizure occurs between the closely fitting plunger and barrel assembly, rather than at the more loosely fitting contact between the gear and plunger. Following seizure, binding and increased friction between the components overcome the effect of the return spring, and the cam follower leaves the cam. As a result, seizure is immediately obvious to the operator. The majority of injector components, including the injector body, were reused following seizure. However, any component damaged by seizure was replaced. The

components automatically replaced include the injector gear, spring, plunger, and bushing assembly.

A schematic diagram of the test loop is shown in Fig. B-2. A low pressure supply pump delivers fuel from the reservoir to the injector. The inlet fuel pressure is adjusted to match that of the vehicle, and the temperature is adjusted to approximately 38°C. High temperature operating conditions are simulated by mounting the injector in a heated block. The temperature of the block is controlled by electric resistance heaters, capable of a maximum operating temperature of 300°C. The test fuel was stored in a 30-gallon container and was filtered prior to use. After passing through the injector, the fuel entered a collection canister and was returned to a separate container. Excess fuel from the injector was cooled using a heat exchanger and returned to the 30-gallon storage tank.

A number of repeat tests were performed to define the potential effects of loading rate on the final applied load required for seizure. These tests were performed using clay treated Jet A-1 at the test conditions defined in TABLE 3. The initial test failed at an applied load of 15 kg after a total test duration of 133 minutes. A second test was performed and the applied load increased at a rate approximately twice that of the first test. The second, more rapid test also failed at 15 kg after a total test duration of 72 minutes. During the remaining tests, the side loading was increased at a rate of approximately 4.54 kg per 10 minutes of testing.

### **C. Test Fuels**

The chemical and physical characteristics of the fuels used during detailed lubricity tests are detailed in TABLE 4. Many of the test fuels are identical to those previously described in References 19, 20, 21, and 23. (Note that these are distinct from the many fuels contained in the fuels database, described in the subsequent section.) The Jet A-1 fuel represents a worst case fuel that combines both low lubricity and viscosity and is known to produce unacceptable wear in full-scale equipment. A fuel equivalent to JP-8 was manufactured from the Jet A-1 by inclusion of 20 PPM of DCI-4A corrosion inhibitor additive, qualified under MIL-I-25017.

**TABLE 4. Principal Characteristics of Test Fuels**

Parameter	Test Method	Test Fuels				
		Jet A-1	JP-8	Cat 1-H	ISOPAR	1% Sulfur
Laboratory No.		19554	20250	19709	20239	19854
Gravity, °API	D 1298	49.5	49.4	34.1	-	31.1
Density, kg/l	D 1298	0.782	0.782	0.8545	-	0.8698
Cloud Point, °C	D 2500	<-50	<-50	-8	-	<-45
Pour Point, °C	D 97	<-50	<-50	-9	-	<-45
Initial Boiling Pt., °C	D 86	160	160	204	-	180
End Point, °C	D 86	218	218	354	-	372
TAN, mg KOH/g	D 974	0.009	0.009	0.08	-	0.16
Sulfur, wt%		0.002	0.002	0.39	0.00	1.02
Flash Point, °C	D 93	44	44	87	49	
Viscosity, cSt/40°C	D 445	1.07	1.07	3.00	3.11	3.36
Aromatics, vol%	D 1319	8.1	8.1	41.0	0.0	33.1
Olefins		0.0	0.0	3.7	0.0	
Cetane Number	D 613	45.0	45.0	48.6	-	44.5
Cetane Index	D976	42.4	42.4	44.8	-	43.0

(Note: True JP-8 contains several other additives that do not affect lubricity.) A number of tests were also performed with higher additive concentrations in Jet A-1. However, it should be recognized that these do not qualify as JP-8 fuels. Cat 1-H and the 1% Sulfur fuel were included as good lubricity, high sulfur fuels.

Several fuels were prestressed prior to wear testing using the STHE. The STHE apparatus was developed in a separate study, a detailed description of which may be obtained in References 1 and 2. The purpose of prestressing the fuel using the STHE is to remove thermally unstable components from the fuel by formation of surface deposits on the heated tube. The mechanism of deposit is temperature sensitive and includes auto-oxidation (below 150°C), thermal oxidative (above 200°C), and pyrolysis on very hot surfaces. The apparatus consists of approximately 70 cm of stainless steel tube immersed in a Techne Fluidized bath model SBL-2D. Prior to each run, test fuel is pumped through the system for 15 minutes to flush the lines of all residue from the previous run or cleanup. The fuel to be stressed is filtered and aerated according to the procedures outlined in ASTM D 3421, the JFTOT test. The test fuel is pumped through the STHE with a standard HPLC pump set to deliver 10 mL/min at a pressure between 800 and

950 psig. The prestressed fuel was then collected and stored in clean glass containers for subsequent wear testing.

In addition to the detailed test data described above, several thousand fuels have been evaluated for commercial sponsors using the Army SLWT. Permission was sought from the original sponsors to include data anonymously in this paper, resulting in a database of approximately 1,200 fuels. This database includes experimental and pilot fuels, as well as those fuels believed to meet EPA and CARB requirements. A number of high-sulfur fuels ( $<0.15$  mass%) are also included as a baseline for comparison. Each of the participating sponsors was requested to provide as much information as possible relating to their fuel. Nonetheless, complete analysis was not available for many of the fuels, reducing the number of data points in some plots. Where possible, ASTM procedures D 4294 (26), D 5186 (27), and D 445 (28) were used to generate sulfur, aromatic, and viscosity data, respectively. However, the data were obtained from a number of sources, and so may contain reproducibility errors between laboratories. All data pertaining to polyaromatics--triaromatics and higher--content were generated at the author's laboratory using a modified version of ASTM D 5186 (27, 29). Results are reported as mass%.

The database may be conveniently subdivided into the following categories (discussed throughout the remainder of the text and also defined in TABLE 5):

- a) "High-Sulfur Fuels" are similar to those historically sold prior to implementation of the 1993 regulations and represent commercially available fuels with a sulfur content greater than 0.15 mass%. Fuels in this category probably do not contain lubricity additives and are used as a baseline for comparison with the newer fuels defined in the sections that follow.
- b) "EPA Fuels" consist of commercially available, low-sulfur fuel samples believed to meet EPA regulations. They have a sulfur content below 0.05 mass% and were obtained throughout the United States (excluding California) following implementation of the 1993 regulations. Some of these fuels may contain lubricity additives.

- c) "CARB Fuels" consist of commercially available, low-sulfur fuel samples believed to meet CARB regulations. They have a sulfur content below 0.05 mass% and were obtained from California following implementation of the 1993 regulations. These fuels must meet the additional requirement mandated by CARB of 10 vol% aromatics or equivalent emissions. Most of these fuels are believed to contain proprietary lubricity additives.
- d) The complete "database" contains many fuels of unknown origin and additive content. Where possible, the fuels are characterized as being either "neat" or "additized." Only those fuels known not to contain additives are characterized as "neat" and are clearly best suited for correlation of lubricity with naturally occurring fuel chemical composition. In contrast, the category "Additized Fuels" contains fuels known to contain additives and also fuels of unknown composition.

As detailed in TABLE 5, the average sulfur content of both the EPA and CARB fuel categories is appreciably below the maximum of 0.05 mass% specified by the EPA. Indeed, approximately one-quarter of the CARB fuels sampled had a sulfur content below 0.01 mass%.

**TABLE 5. Summary of Fuels Database**

Property	Category			
	High-Sulfur Fuels	EPA Fuels	CARB Fuels	Complete Database
<b>Sulfur, mass%</b>				
Minimum	0.15	0.01	0.001	0.000
Mean	0.305	0.0313	0.021	0.036
Maximum	0.45	0.05	0.047	0.728
<b>Aromatics, mass%</b>				
Minimum	18.1	0.01	5.1	0.0
Mean	31.5	28.0	21.1	16.1
Maximum	44.0	42.0	39.0	65.0



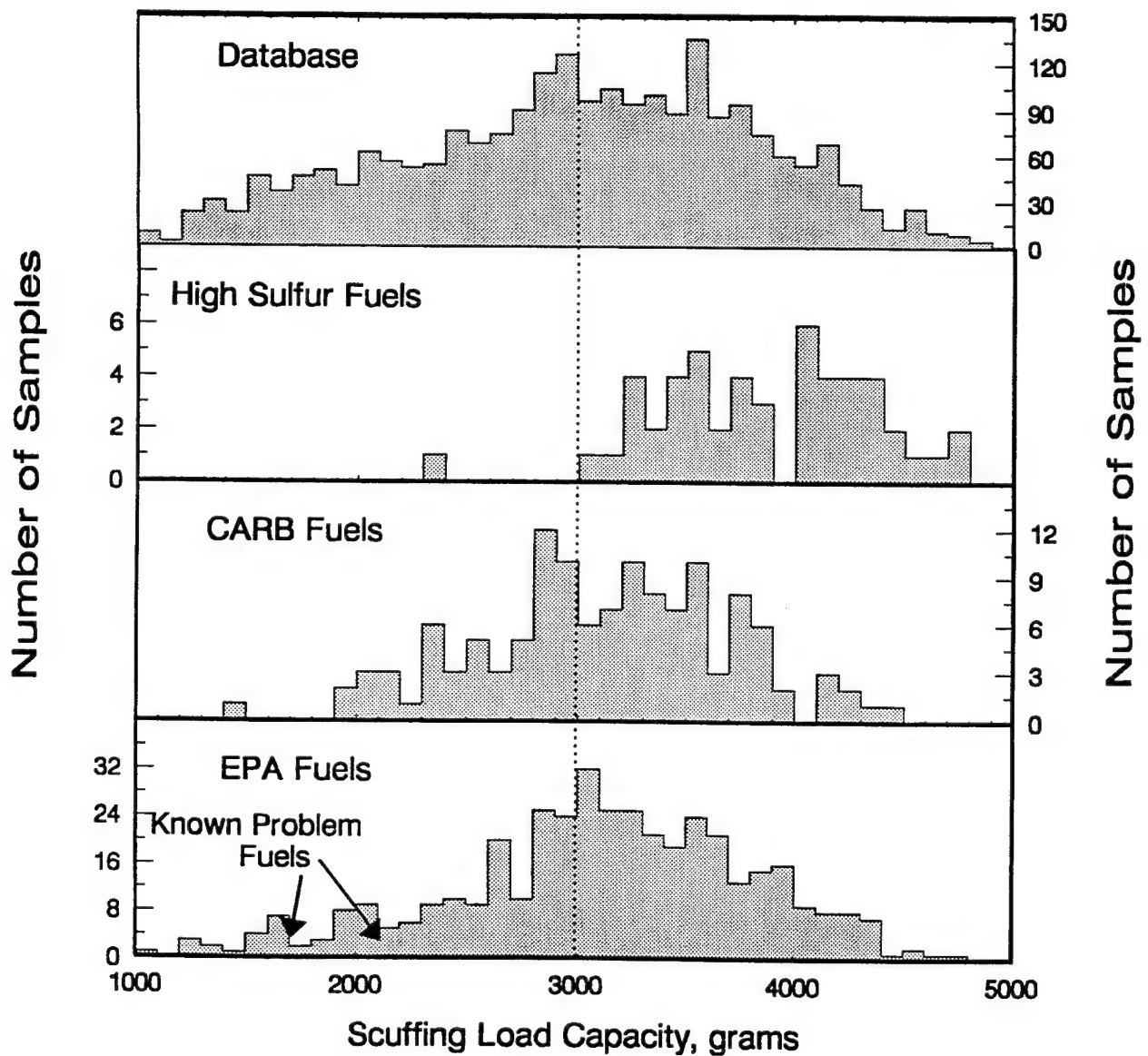
## **D. Summary of Laboratory Wear Test Database**

### **1. Effect of 1993 Federal and State Fuel Specifications on Fuel Lubricity**

Graphs summarizing the test results obtained using the Army SLWT are shown in Fig. 1. Four separate histograms show the distribution of results for each of the data categories described in TABLE 5. The uppermost histogram is for the complete database. The distribution of results is between 1,000 and 5,000 grams minimum applied load, with a mean of approximately 3,000 grams; however, this distribution of data is probably not representative of commercially available fuels.

The remaining three histograms in Fig. 1 show results for high-sulfur, CARB, and EPA fuels, all of which are commercially available. As expected, most of the high-sulfur fuels produced a scuffing load capacity above 3,000 grams, which is believed to be the minimum result necessary to provide long-term equipment durability. By comparison, a relatively large number of both the CARB and EPA fuels produced results that fall below the 3,000-gram durability minimum. Furthermore, several fuels produced results below 2,000 grams, which corresponds to very poor lubricity. The tests for several of the lower lubricity fuels were obtained (but not plotted) in duplicate to minimize the possibility of error with these more critical fuels.

Three of the lowest lubricity fuels, as measured using the Army SLWT, were reported to have reduced equipment durability at three separate military bases.<sup>(5)</sup> The results for these fuels are highlighted in black in Fig. 1, and the remaining characteristics are summarized in TABLE 6. The fuel injection system failure rates recorded at one of the military bases is summarized in Fig. 2 on a month-by-month basis. Samples of the high-sulfur fuel used by this base previous to September 1993 are not available but can be assumed to be of good lubricity. Failure rates approximately doubled following September 1993, which corresponds to the introduction of the low lubricity Fuel C, as described in TABLE 6. The base has subsequently reported that a number of replacement pumps have failed for the second time.



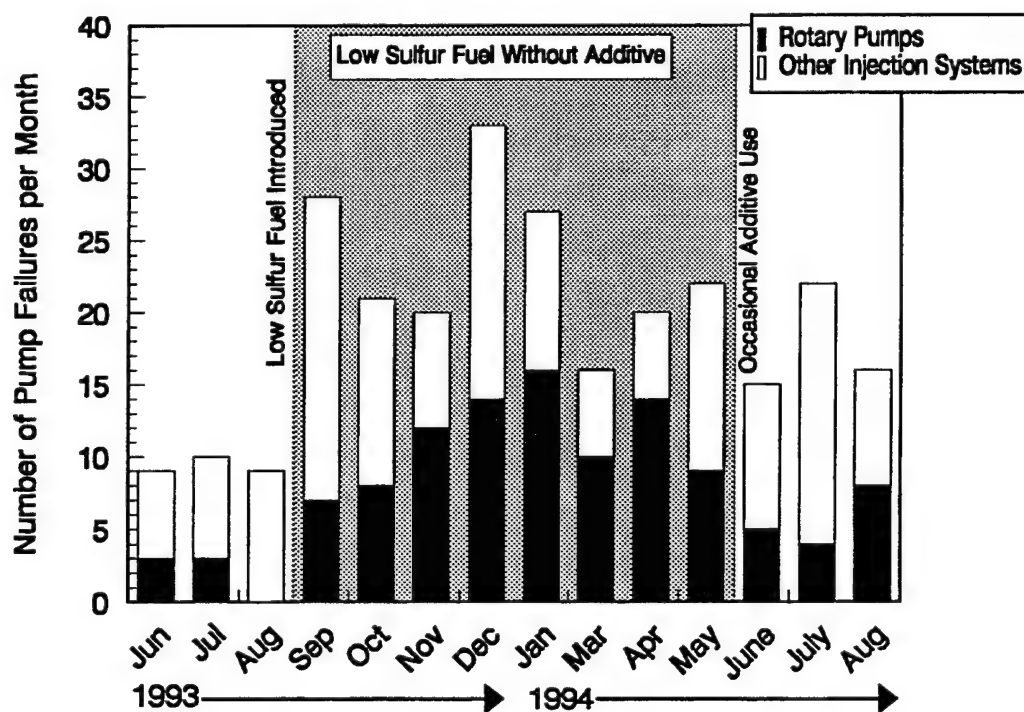
(Note: The High-Sulfur, CARB, and EPA fuel categories each represent commercially available fuels.)

**Figure 1. Histograms summarizing test results obtained using the Army SLWT for the fuel categories described in TABLE 5**

**TABLE 6. Physical and Chemical Characteristics of Fuels Suspected to Have Promoted Premature Fuel System Failures at Military Bases**

Military Base	Army SLWT Results, grams	Sulfur, mass%	Aromatics, mass%	Viscosity, cSt at 40°C
A	1,700	0.01	22.0	1.28
B	2,100	0.02	13.5	1.63
C	1,600	0.04	22.5	1.44

The failure rate was marginally reduced in June 1994 by the addition of 250 mg/L of a lubricity additive based on di-linoleic acid that increased the scuffing load capacity to 3,000 grams from its original level of 1,600 grams. Significantly, each of the fuels has combined low lubricity and viscosity below 1.6 cSt at 40°C, minimizing both boundary and hydrodynamic lubrication mechanisms.



(Note: Low lubricity fuel introduced September 1994.)

**Figure 2. Pump failure rates recorded at a military base by month**

## 2. Correlation of Fuel Lubricity With Chemical Composition

It is unlikely that a perfect correlation between chemical composition and lubricity will be easily derived, considering the multitude of surface-active compounds potentially present in each fuel. In addition, some aspects of fuel lubricity are sensitive to concentrations as low as 20 parts per million (ppm). Nonetheless, a broad correlation between lubricity and refining severity, as indicated by sulfur and/or aromatics content, would be useful. This section evaluates the complete fuels database, including additized and experimental fuels. The neat fuels are plotted separately, and the coefficient of determination ( $R^2$ ) is summarized in TABLE 7.

**TABLE 7. Summary of Regression Analysis for the Army SLWT**

Parameter	Regression Type	Coefficient of Determination ( $R^2$ )	
		Neat Fuel	Additized
Full-scale equipment*	Linear	0.66**	0.66**
ASTM D 5001	Geometric	0.57**	0.57**
Viscosity	Linear	0.44	0.24†
Density	Linear	N/A‡	0.21†
Sulfur	Linear	0.26	0.06†
Total aromatics	Linear	0.64	0.16†
Diaromatics	Geometric	0.84	--
Polyaromatics§	Geometric	0.69	--

\* Data applies to four different injection equipment types tested at five laboratories. Considerably improved correlation is produced within each equipment category.

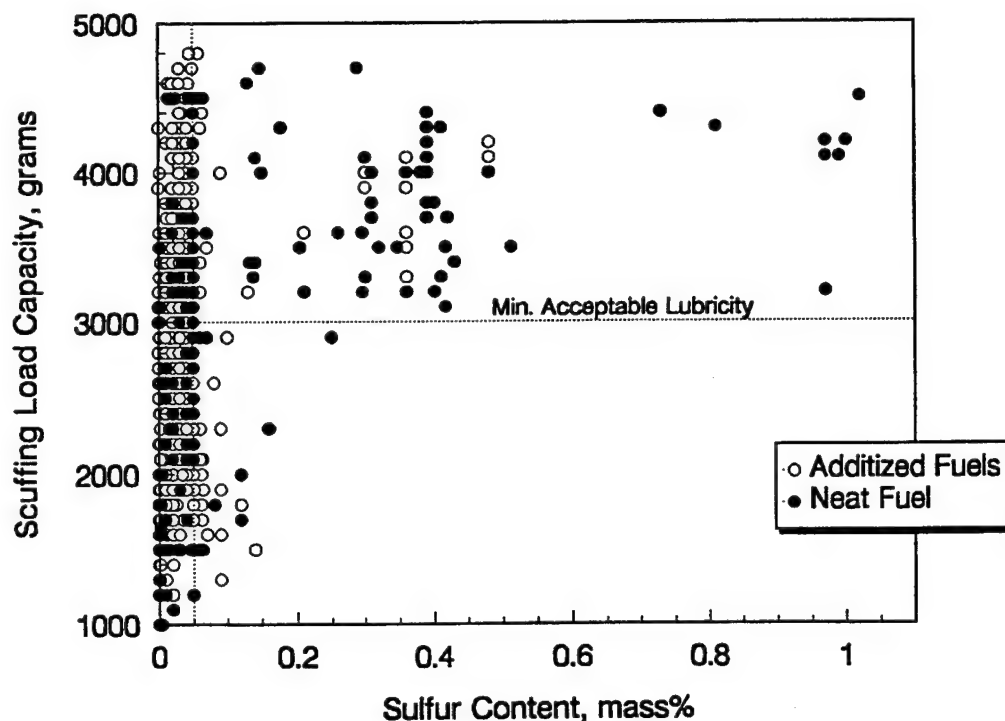
\*\* Data applies to both neat and additized fuels.

† Correlation degraded by additives.

‡ N/A = Not available

§ Triaromatics and higher

Increasingly severe hydrotreatment will initially reduce sulfur compounds, followed by the higher molecular weight aromatics. The correlation between fuel sulfur content and scuffing load capacity is shown in Fig. 3. No direct correlation exists between sulfur content and fuel lubricity. In particular, unadditized low-sulfur fuels had variable lubricity, ranging from very good to bad,

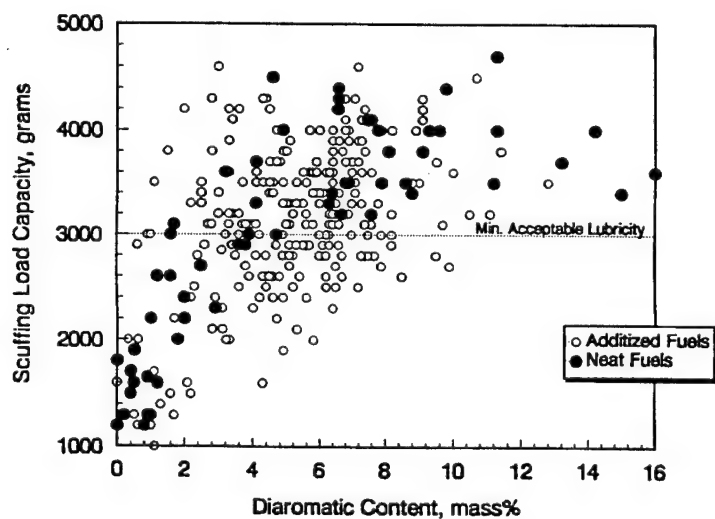
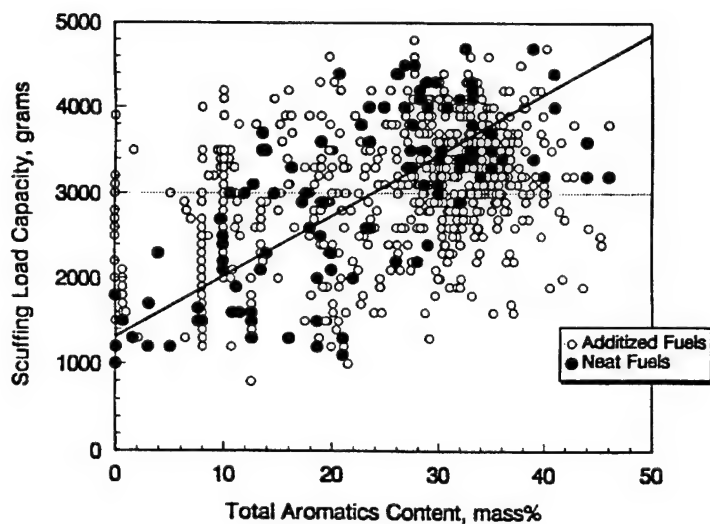


**Figure 3. Correlation between scuffing load capacity and sulfur content for the complete database, including noncommercially available fuels**

resulting in a low coefficient of determination. As a result, it is impossible to differentiate among post-1993, low-sulfur fuels on this basis alone. Significantly, no fuel with a sulfur content above 0.15 mass% (i.e., typical pre-1993, high-sulfur fuels) showed poor lubricity.

Scuffing load capacity is plotted as a function of total aromatics content in Fig. 4a. Some directional correlation exists, with coefficients of determination of 0.64 and 0.16 for neat and additized fuels, respectively. However, very high total aromatics content is necessary to ensure good lubricity. Conversely, no fuel with an aromatics content below 10 mass% (that did not contain a lubricity additive) had good lubricity. The correlation between fuel lubricity and diaromatic content is shown in Fig. 4b, while Fig. 4c illustrates the correlation between fuel lubricity and polyaromatic content. Relatively good (nonlinear) correlation is present in both instances, with a maximum coefficient of determination of 0.84, as detailed in TABLE 7. In general, additives effectively improved poor lubricity fuels that contained smaller amounts of di- and polyaromatic compounds. However, the additives appear to produce significantly less improvement in better lubricity fuels.

a. Total aromatics content



b. Diaromatic content

c. Polyaromatic content

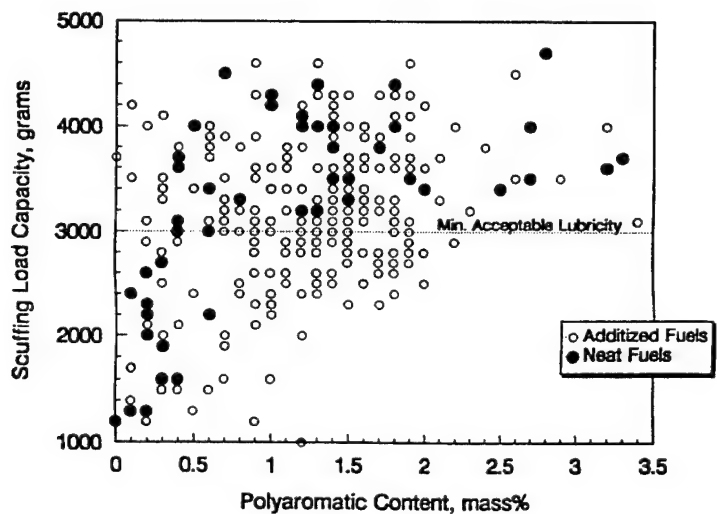


Figure 4. Correlation between scuffing load capacity and aromatics content for various aromatics types, including noncommercially available fuels

Tests were performed using the Army SLWT procedure to define the effect of 26 compounds on lubricity, with the results shown in TABLE 8. Mono- and diaromatic compounds were added at 5 mass%, and polyaromatic and olefin compounds were added at 1 mass%. Organo-sulfur compounds were added at the lower concentration of 0.5 mass% to reflect their lower concentration in real fuels. None of the polyaromatic compounds were completely soluble at this concentration, and they were mixed with 2 vol% toluene, which improved solubility somewhat. It should be recognized that these compounds only represent a small fraction of all possible sulfur and aromatic components present in real fuels.

Most of the compounds produced a marginal increase in the scuffing load capacity of the fuel, normally becoming more effective as molecular weight increased. However, none of the compounds increased scuffing load capacity to the extent predicted by consideration of total sulfur and aromatic content in real fuels, as shown in Figs. 3 and 4. It is likely that sulfur and aromatics compounds simply reflect the effects of refinery severity on other surface-active compounds in the fuel.

### **3. Correlation With Physical Parameters**

Full-scale fuel injection system equipment is sensitive to viscosity, and many manufacturers specify a minimum viscosity of 1.8 cSt at "operating temperature." The Army SLWT was designed to measure boundary lubricating characteristics that depend only on chemical composition. The effects of viscosity are minimized through use of a counterformal contact geometry, as well as the naturally low viscosity of most fuels. Nonetheless, Fig. 5 indicates that low viscosity fuels tend to have a slightly lower scuffing load capacity, with a coefficient of determination of 0.44 and 0.24 for neat and additized fuels, respectively.

This effect is partially due to hydrodynamic lift between the specimens during testing, which is always present during lubricated sliding. However, chemical and physical characteristics are related, particularly for straight-run distillate fuels. (Processes such as solvent extraction, clay treatment, or catalytic hydrogenation are capable of producing clean, low lubricity fuels with high

**TABLE 8. Effect of Various Hydrocarbon Compounds on Lubricity**

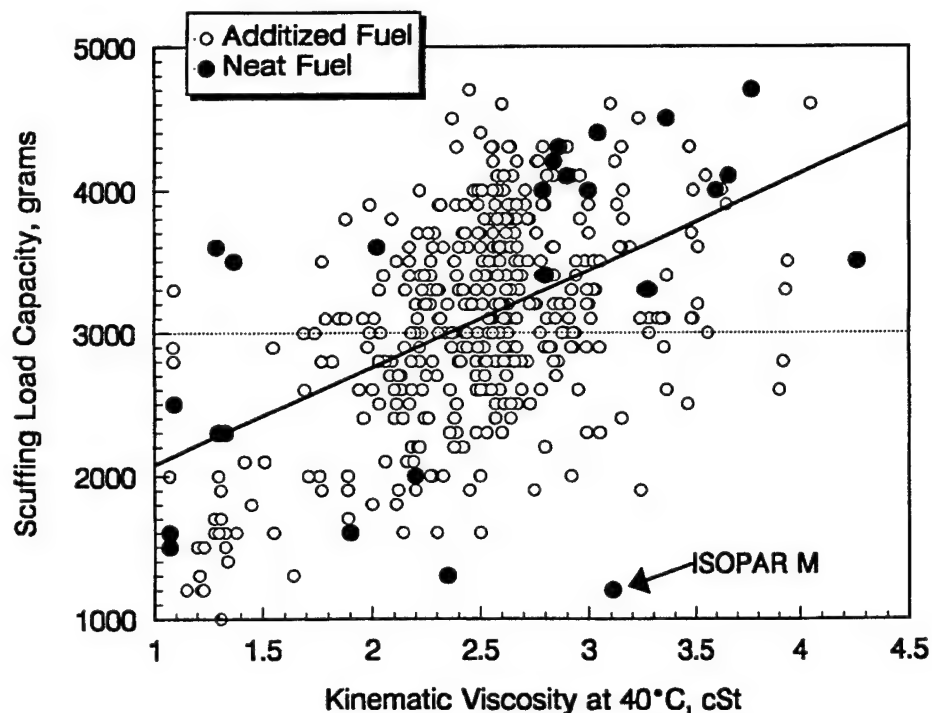
Compound Type	Molecular Weight	Concentration, mass%	Improvement, grams*
<b>Organo Sulfur</b>			
Phenylsulfide	154	0.5	300
Butyldisulfide	178	0.5	300
Benzylphenylsulfide	200	0.5	300
Phenyldisulfide	218	0.5	600
Benzylidisulfide	246	0.5	300
<b>Monoaromatics</b>			
Toluene (Methylbenzene)	92	5	0
Xylene (Dimethylbenzene)	106	5	200
S-Butylbenzene	134	5	600
N-Tetradecylbenzene	214	5	800
<b>Diaromatics</b>			
Naphthalene	128	5	0
1,2,3,4-Tetrahydronaphthalene	132	5	500
Methylnaphthalene	142	5	500
1,4-Dimethylnaphthalene	156	5	700
Dibenzothiophene	184	5	700
Dinonylnaphthalene	380	5	500
<b>Polyaromatics</b>			
Anthracene**	178	1	0†
Pyrene**	202	1	100
1,2-Benzanthrene**	216	1	400
1,2-Benzopyrene**	252	1	100†
<b>Olefins</b>			
Cyclohexene	82	1	0
Octene	112	1	300
Nonene-I	126	1	800
Dicyclopentadiene	132	1	200
Decene-I	140	1	1100
Tetradecene	198	1	1100
1-Octadecene	252	1	100

\* The improvement represents the change in lubricity caused by inclusion of the additive in a neat Jet A-1 fuel which contains 0.002 mass% sulfur and 8 mass% aromatics, with a scuffing load capacity of 1,200 grams.

\*\* Contains 2 vol% toluene to assist in dissolution.

† Incomplete dissolution of additive may affect results.





**Figure 5. Correlation between kinematic viscosity and scuffing load capacity for the complete database, including noncommercially available fuels**

viscosity.) As a result, a similar but very slight directional trend may be observed between scuffing load capacity and density, as shown in Fig. 6, with a coefficient of determination of 0.21. Density is unlikely to have any direct effect on the lubrication process. However, higher density, higher boiling point fuels tend to have a correspondingly higher sulfur and aromatics content, as well as higher viscosity (30), all of which have been shown to be related to scuffing load capacity.

A wide range of both commercially available and experimental lubricity additives has been evaluated using the Army SLWT. In general, the response obtained from lubricity additives depends on both additive concentration and the lubricity of the base fuel. The combined effects of both variables were plotted using a wear mapping procedure similar to that developed in Reference 19. The increase in scuffing load capacity produced by lubricity additives is plotted in Fig. 7 as a simultaneous function of additive concentration and the scuffing load capacity of the fuel without additive. (The increase in scuffing load capacity is defined as the result obtained for the additive-containing fuel minus the result obtained for the neat fuel.)

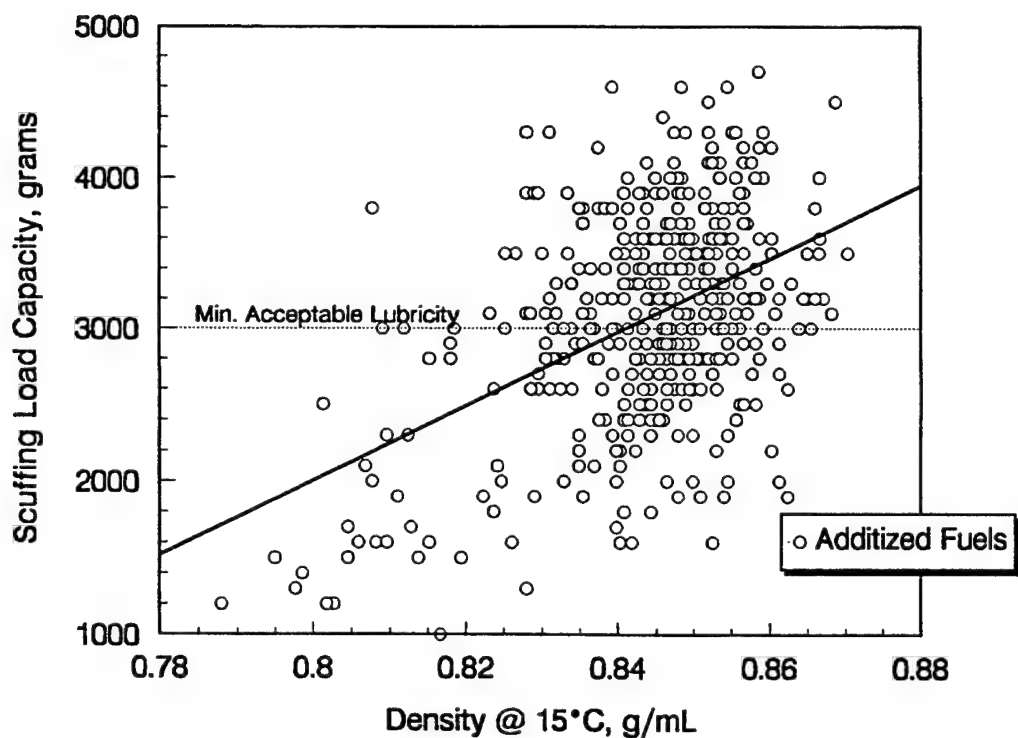


Figure 6. Correlation between density and scuffing load capacity for the complete database, including noncommercially available fuels

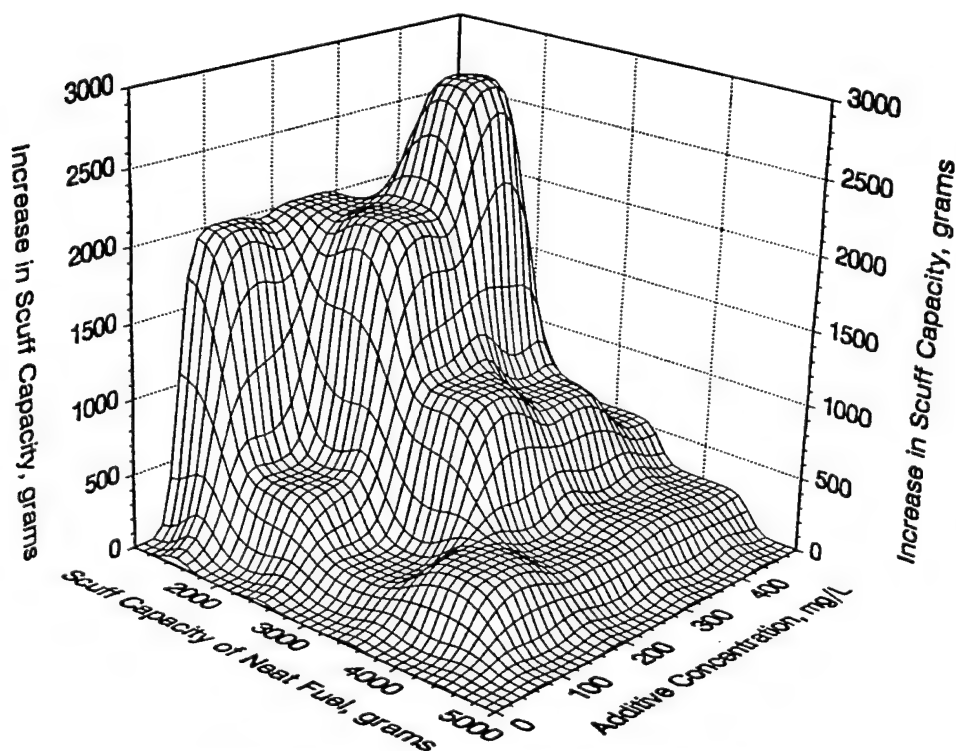


Figure 7. Increase in scuffing load capacity due to additives plotted as a simultaneous function of additive concentration and the scuffing load capacity of the neat fuel

Figure 7 was generated from approximately 400 fuel and additive combinations. Most of the additives used are commercially available, although some experimental compounds are also present. Ineffective additives, which were believed to be experimental in nature, were excluded from the database (it is assumed that they will not be made commercially available). The plotted data has been smoothed (filtered) by averaging neighboring data points. The step transitions on the surface are the result of the smoothing process. Clearly, Fig. 7 only reflects the broad trends observed and does not imply that all additives fit this description. Moreover, the filtering process masks variations in additive response in differing fuel compositions, as well as variations in additive effectiveness. However, most additives produced surprisingly similar results, and in general, additive effectiveness increases with concentrations up to approximately 100 to 200 mg/L. Further increases in concentration up to approximately 400 mg/L only marginally increase wear resistance. Additive concentrations of 1,000 and 2,000 mg/L are widely used, but typically provide no additional protection (these concentrations are not plotted in Fig. 7). These higher concentrations are normally found with multifunctional additive packages, which contain a number of additives dissolved in a carrier fluid to facilitate blending at the vehicle.

The additive concentration required to increase the scuffing load capacity of a given fuel is considerably greater than that needed to eliminate the oxidative corrosion wear mechanism which predominates in jet kerosene fuels and is measured using the test procedure defined in ASTM D 5001.<sup>(21)</sup> Additive concentrations specified in MIL-I-25017 for military aviation fuels typically range from 9 to 22.5 mg/L.<sup>(31)</sup> Such concentrations successfully eliminate surface oxidation without providing a strong boundary layer and show little or no improvement in scuffing wear resistance. The composition of most additives evaluated in the present study is unknown. Significantly, however, the increase in scuffing load capacity plotted in Fig. 7 is typical of that observed for high concentrations of simple di-carboxylic acid compounds, such as di-linoleic acid (curves for which may be found in Reference 23), normally used as corrosion inhibitors in additives qualified under MIL-I-25017.

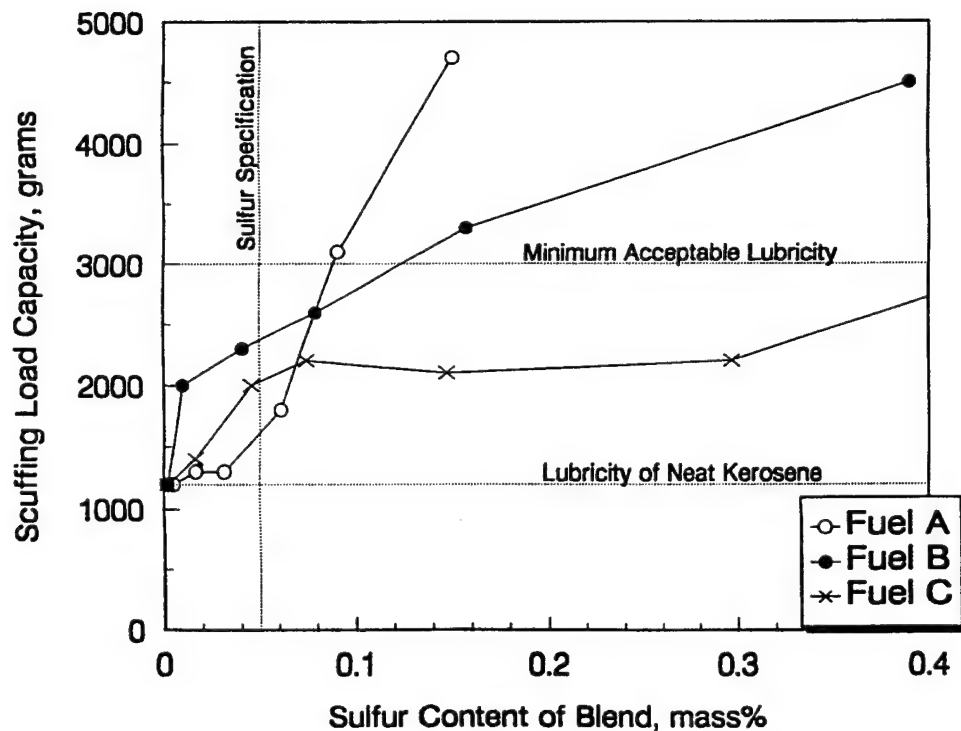
The benefits provided by the lubricity additives are also sensitive to fuel composition, with the poorest lubricity fuels experiencing the greatest improvement. In general, the optimum additive concentration increased the scuffing load capacity of the fuel to approximately 3,500 grams,

irrespective of the initial lubricity of the base fuel. This corresponds to the lubricity of a typical, high-sulfur fuel and may normally be achieved using a high concentration of a di-carboxylic acid. However, a number of exceptions were observed in which the effectiveness of a single additive was found to vary greatly between two fuels of similar initial lubricity.

#### **4. Alternate Fuel Blends**

A number of alternate solutions for low lubricity other than traditional lubricity additives have been evaluated at TFLRF. For example, blending a small volume of good lubricity fuel with low lubricity fluid provides a significant improvement in wear resistance. For many fuel streams, the level of hydrotreatment necessary to reduce aromatics content below 10 to 15 vol% virtually eliminates sulfur from the final product. As a result, some back-blending of a high-sulfur feedstock with the final product may occur without exceeding the 0.05 mass% sulfur limit and with only a marginal effect on aromatics content.

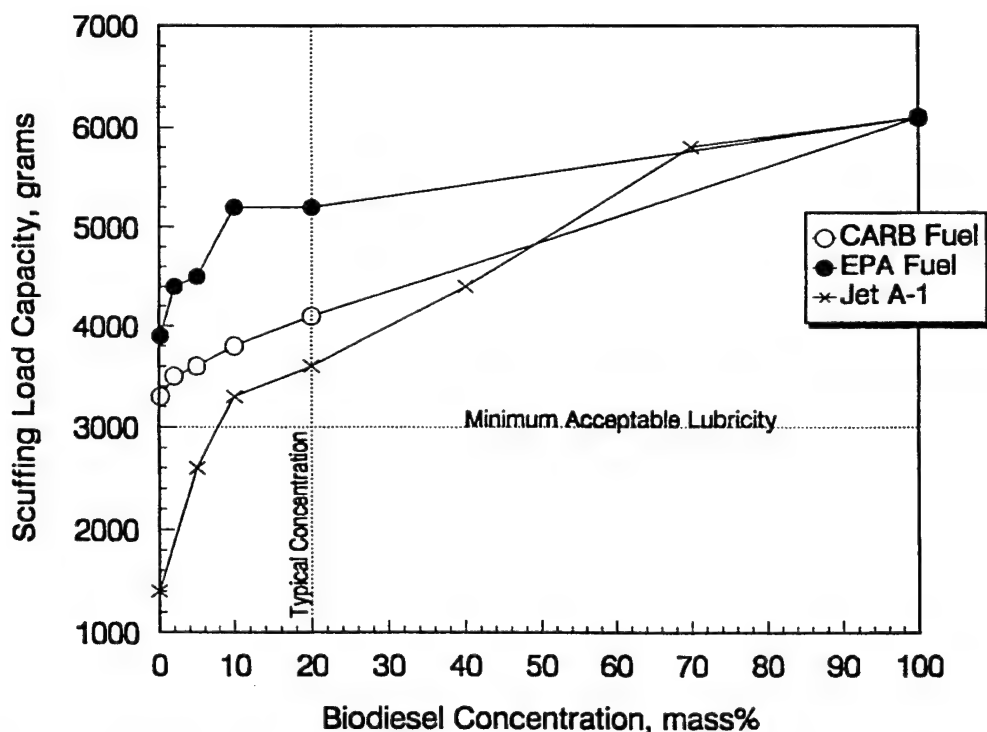
The effects of blending different high-sulfur diesel fuels with a neat Jet A-1 kerosene fuel are shown in Fig. 8. Three fuels were selected with sulfur contents of 0.15, 0.39, and 0.72 mass% sulfur, designated as A, B, and C, respectively. Fuel A is particularly effective, and the lubricity of the Jet A-1 fuel is improved by approximately 1,200 grams when the combined sulfur content of the blend is 0.05 mass%. It should be noted that the Jet A-1 base fuel is of especially low lubricity and initially contains only 0.002 mass% sulfur. This increase would significantly improve most normal fuels. Indeed, an increase of 1,200 grams would be sufficient to improve the lubricity of almost all the commercially available fuels in Fig. 1 to the 3,000-gram minimum lubricity level. However, fuels B and C are less effective in improving lubricity at similar total sulfur concentrations. Conversely, kerosene fuels are commonly blended with higher viscosity fuels to allow their use in arctic conditions. In this instance, dilution with kerosene fuel will produce a marked decrease in overall lubricity.



(Note: The right-most point on each curve corresponds to neat high-sulfur fuel.)

**Figure 8. Effect of blending high sulfur fuels on the scuffing load capacity of a low lubricity, low sulfur aviation kerosene fuel**

Alternative fuels, such as the methyl ester of soybean oil (biodiesel), act as a low emission component for exhaust emissions reduction. The effects of biodiesel on the lubricity of an EPA fuel and a CARB fuel are plotted in Fig. 9. The biodiesel fuel consisted of soybean oil transesterified with methanol to form methyl ester and had exceptionally good lubricity. The scuffing load capacity of the neat biodiesel was in excess of 6,000 grams, which is superior to any diesel fuel evaluated to date. Both the EPA and CARB base fuels had good initial lubricity, which was further improved through blending with biodiesel. In the United States, fuels derived from soybean oils are normally blended into diesel fuels at a concentration of 20 vol%. This treatment rate increased the performance of even the very poor lubricity Jet A-1 fuel to an acceptable level.



**Figure 9. Effect of blending methyl ester (soydiesel) on the scuffing load capacity of good lubricity, commercially available diesel fuels**

#### **5. Effect of Removing Thermally Unstable Fuel Components by Prestressing**

It is generally accepted that during fuel-lubricated sliding, oxygen reacts with the surface metal to form an oxide layer.<sup>(21)</sup> The corrosive action continues as the oxide layer is removed by abrasion and a new oxide layer is formed. Polar compounds in the fuel are believed to adsorb on the metal surface and shield the surface from oxidative corrosion. However, other fuel properties such as hydrocarbon type have also been found to affect the wear process, and it has been proposed that other effects are also present. For example, Benchaita et al <sup>(32)</sup> found that saturated hydrocarbons produced less wear than aromatics. Significantly, substituted alkyl aromatics auto-oxidize much more readily than alkanes. These observations have led to the theory that the wear process also depends on the oxidative stability of the fuel.

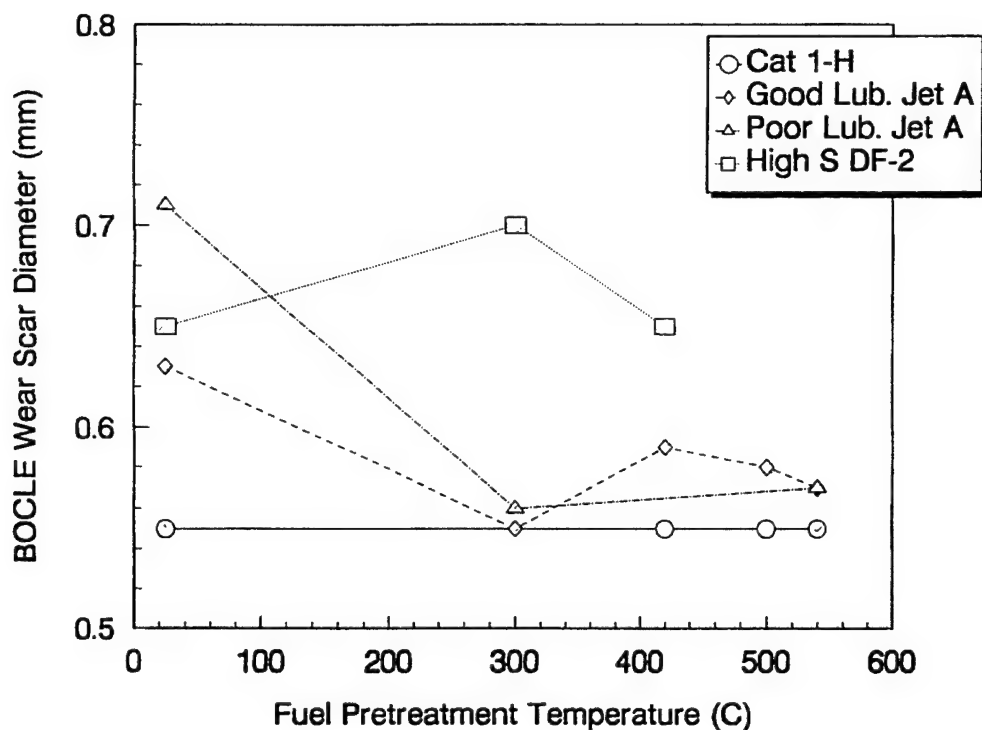
Another clue that the mechanism involves fuel oxidation is that high molecular weight hydrocarbon deposits are often formed around the wear scar in the BOCLE test. The deposits have not been thoroughly characterized, but they are not unlike the gummy substances formed from the auto-oxidation of fuel and the harder fuel deposits produced in thermal stability tests.

Hsu et al (33) predict temperatures in the neighborhood of 375°C at asperity contact points formed during sliding wear. At such high temperatures, fuel oxygen is consumed by auto-oxidation reactions in a millisecond time frame. It appears that the fuel temperatures associated with wear are similar to those in thermal stability tests and fuel pretreatment using the single tube heat exchanger.

The auto-oxidation theory seems to give a more plausible explanation of the effects of fuel properties on wear. When fuels auto-oxidize, the first step in the reaction is the formation of organic hydroperoxides. Some of the hydroperoxides decompose into alcohols and aldehydes, which oxidize further and become carboxylic acids. Hydroperoxides are powerful oxidizing agents which may readily oxidize metal surfaces. Carboxylic acids react with metal oxides to form fuel-soluble organic salts. Thus, the action of the hydroperoxide and carboxylic acid working hand-in-hand is a much more corrosive vehicle than dissolved oxygen in the fuel. More detailed research is currently in progress to verify the relationship between thermal degradation and wear.

In the present study, laboratory-scale wear tests were performed with four fuels that had been prestressed using the single tube heat exchanger. Prestressing removes the least thermally stable components from the fuel prior to wear testing and so should affect the reaction process present during wear. Cat 1-H, high sulfur DF-2, and both clay-treated and untreated Jet A-1 fuels were each prestressed at 300, 420, 500, and 540°C. The chemical and physical characteristics of each fuel prior to prestressing using the STHE may be found in TABLE 4.

Previous studies have shown that the ASTM D 5001 BOCLE test is especially sensitive to the effects of oxidative corrosion wear.(21) In addition, this test is especially sensitive to trace concentrations of surface active compounds. The results of laboratory-scale wear tests performed according to ASTM D 5001 are plotted in Fig. 10 as a function of fuel pretreatment temperature using the STHE. Clearly, the untreated fuels have widely varying initial lubricity. The Cat 1-H fuel has best initial lubricity and is completely unaffected by prestressing using the STHE. This is to be expected, as wear with high sulfur fuels is largely unaffected by oxidative corrosion.



**Figure 10. Effect of fuel pretreatment temperature in the STHE on lubricity measured using the ASTM D 5001 wear test**

Similarly, trace concentrations of surface active compounds have no effect on the already complex chemistry present.

In contrast, the Jet A-1 fuel has lower initial lubricity than Cat 1-H but is significantly improved by prestressing. These fuels have few natural corrosion inhibitors and typically wear by an oxidative mechanism that is sensitive to the availability of both oxygen and moisture.<sup>(21)</sup> Moreover, wear with these very pure kerosene fuels is highly sensitive to trace concentrations of chemically reactive compounds. It would appear that prestressing of the fuel is creating surface active species.

The high sulfur DF-2 fuel has surprisingly low lubricity, as measured using the ASTM D 5001 wear test. Indeed, both the new and prestressed fuel had wear scar diameters of 0.65 mm or greater. Subsequent tests performed using the Army SLWT indicate that this fuel has excellent load carrying ability and scuffing wear resistance. The ASTM D 5001 procedure is a lightly loaded wear test, most sensitive to oxidative corrosion. It is likely that the 1 mass% sulfur content of the present fuel is creating a related surface corrosion mechanism due to chemical



reaction of the fuel sulfur within the contact area. Post-test examination of the wear scar using an optical microscope shows a polished topography typical of a corrosive mechanism. In any instance, wear with the high-sulfur fuel is not reduced by prestressing using the STHE.

Laboratory wear tests were performed as a function of applied load using the Cameron-Plint wear test apparatus. The results obtained with Cat 1-H fuel that had been pretreated using the single tube heat exchanger are plotted in Fig. 11. The test conditions are defined in TABLE 2. High temperature fuel prestressing produced only a slight increase in the wear rate observed at room temperature, which reflects the previous results obtained in BOCLE tests performed according to ASTM D 5001. At higher loads, a sharp increase in wear is observed, due to the onset of adhesive scuffing. This transition point reflects the load carrying ability (or scuffing load resistance) of the fuel. Clearly, thermal prestressing reduced the load carrying ability of Cat 1-H fuel by up to 30 percent.

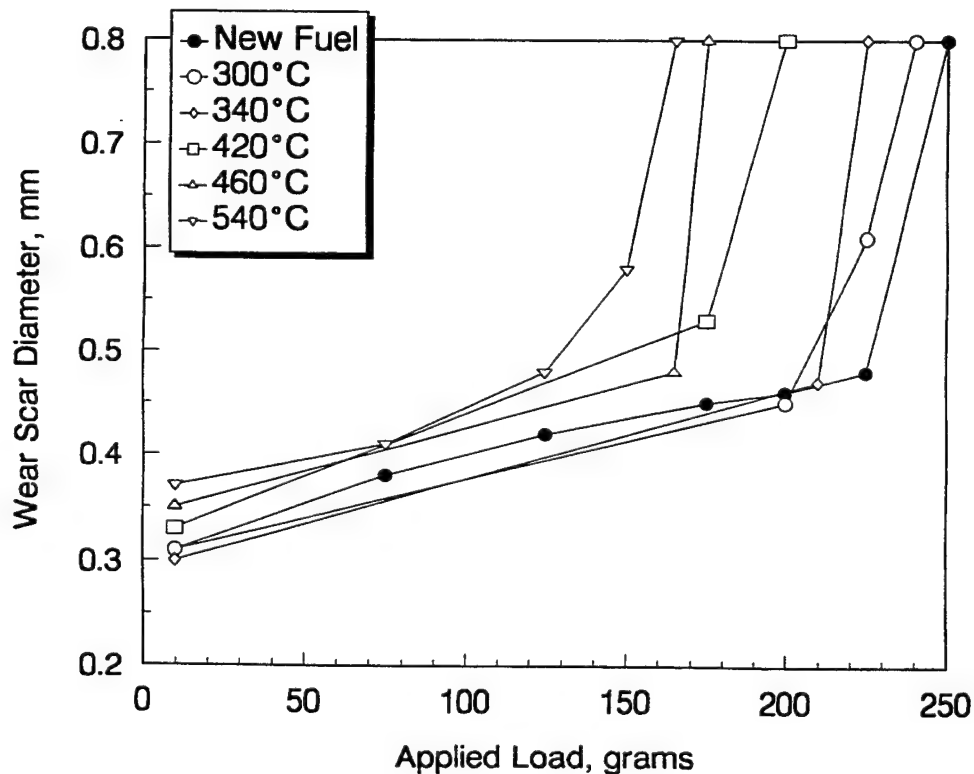


Figure 11. Effect of fuel pretreatment using the STHE on wear resistance of Cat 1-H as measured using the Cameron-Plint apparatus

The applied load required for the onset of adhesive scuffing was defined for neat and clay filtered Jet A-1, and high-sulfur diesel. The results obtained are plotted in Fig. 12, along with those previously discussed for Cat 1-H. The scuffing load capacities of high-sulfur diesel and Jet A-1 fuels are almost independent of thermal prestressing. The slight variations observed are likely to be due to test repeatability. The scuffing load result for Jet A-1 is in direct contrast to the low load wear test results, plotted in Fig. 10.

## 6. Effect of Operating Temperature

The results obtained in the previous sections indicated that fuel lubricity is affected by composition and by thermally prestressing the fuel at temperatures above 300°C. However, the preceding data were obtained from wear tests performed at room temperature, which is not normally representative of real world conditions. The operating temperature in many current applications will be greatly in excess of 80°C, depending on equipment type. Future applications in partially cooled military equipment are certain to be higher.(34)

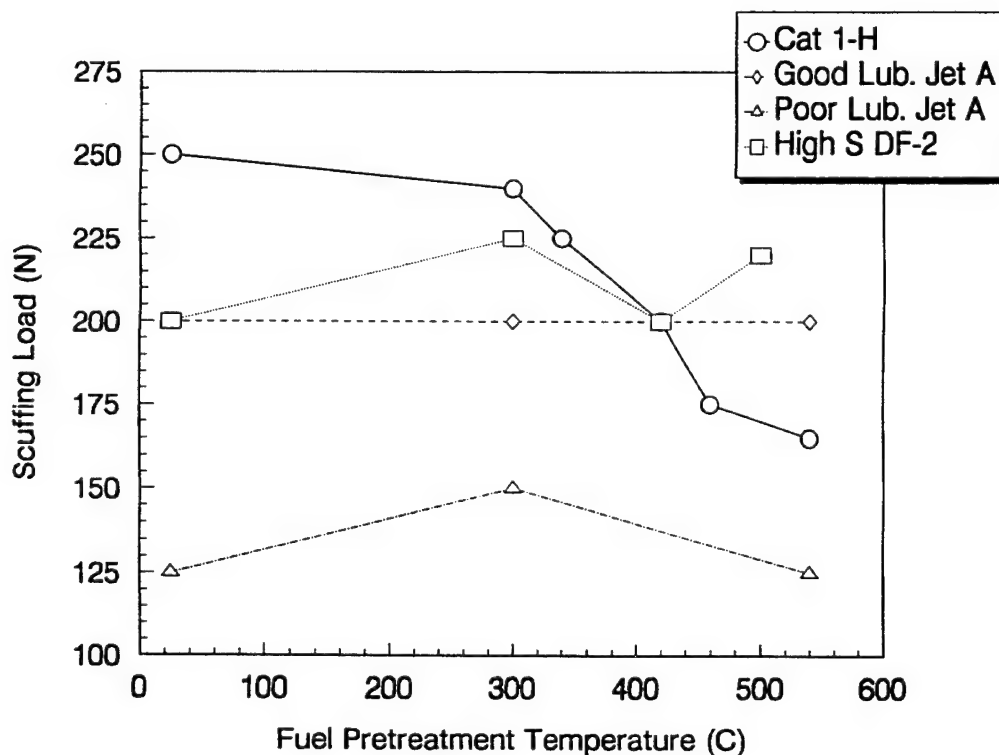


Figure 12. Effect of fuel pretreatment using the STHE on scuffing load capacity of various fuels, as measured using the Cameron-Plint apparatus

The effects of increasing temperature on the results of the ASTM D 5001 BOCLE test are shown in Fig. 13. The BOCLE tests were carried out at temperatures between 0° and 90°C with clay treated Jet A-1 and Jet A-1 containing 20 PPM DCI-4A, which is effectively equal to JP-8. (JP-8 contains several other additives that do not affect lubricity.) As the tests were not performed at 25°C, they deviate from the ASTM test standard. In addition, the fuel was not aerated before each test, as is specified in the standard. This variation helped minimize loss of lighter fractions from the fuel, which has a flash point of 44°C.

Erratic test results were achieved for clay-treated Jet A-1 at temperatures above approximately 50°C. The predictable curves exhibited by the additized fuels were not apparent. This effect may be due to competition between increased rates of fuel oxidation and surface corrosion at high temperatures. Fuel oxidation reactions form oxygenated species (i.e., carboxylic acids, aldehydes, alcohols, etc. that act as good lubricity agents because of their polar nature). In general, however, the wear rate with neat Jet A-1 decreased with increasing temperature, while

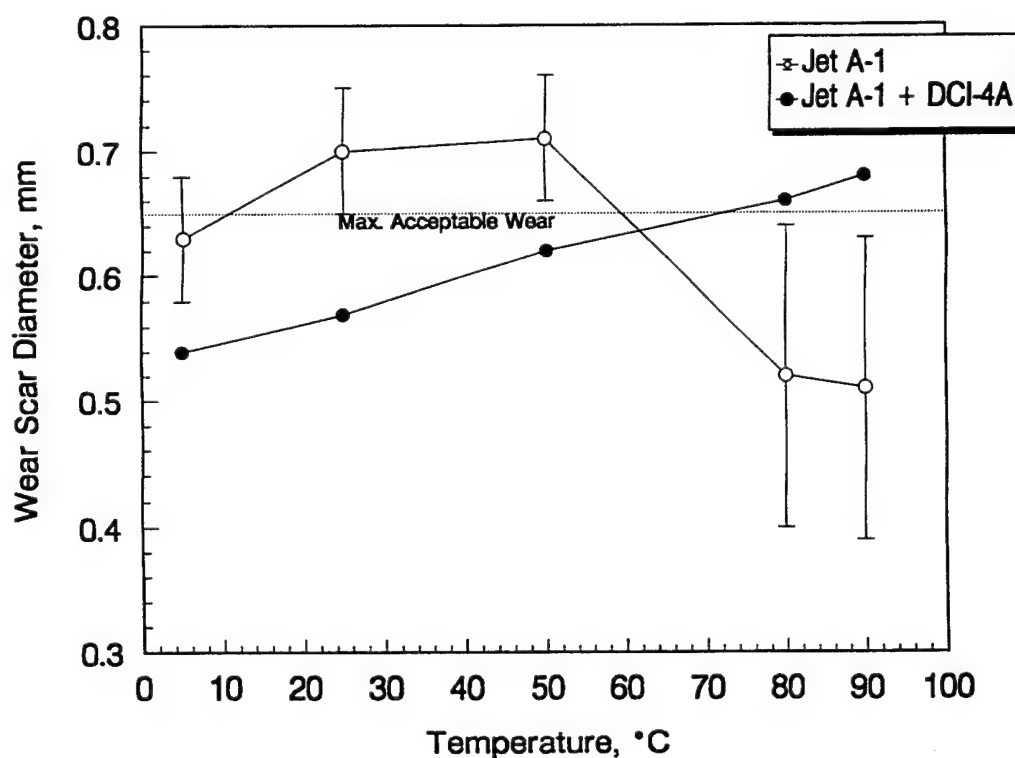


Figure 13. Effect of operating temperature on the ASTM D 5001 BOCLE test

the wear rate with Jet A-1 that contained DCI-4A increased with temperature. The decrease in additive effectiveness is probably due to desorption of the weakly bonded dilaoleic acid molecules, combined with decreasing hydrodynamic lift.

Laboratory wear tests were performed with clay-treated Jet A-1 using the Cameron-Plint apparatus. The tests were performed over a wide range of temperatures, from ambient to 100°C at various applied loads. The specimens are completely immersed in the fuel reservoir during the test. However, unlike previous tests, the high temperature fuel sample was continuously drained and replaced. Continuous flow-through of fresh fuel occurs in real applications and also minimizes the effects of evaporation in the open wear test system. A relatively low flow rate was used to ensure a stable test temperature.

Figure 14 shows the wear map produced by Jet A-1 fuel, plotted as a simultaneous function of applied load and temperature. Clearly, wear is sensitive to fuel test temperature. In particular,

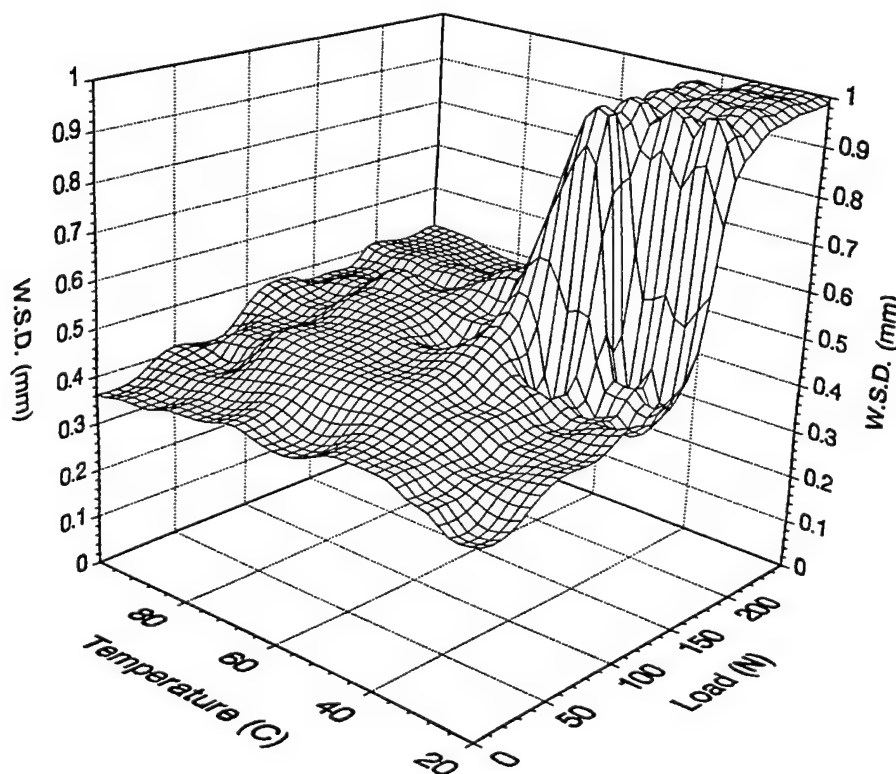


Figure 14. Wear map for AISI E 52100 steel lubricated with Jet A-1

the applied load required for the onset of adhesive scuffing is increased at high temperatures. However, the laboratory-scale wear tests are largely insensitive to the effects of viscosity, which decreases with temperature. Most real applications (such as unit fuel injectors) rely on elastohydrodynamic and hydrodynamic lubrication and are likely to have a more complex relationship with temperature.

## 7. Results From Full-Scale High Temperature Unit Injector Tests

Full-scale unit injector tests were performed with Jet A-1, Cat 1-H, and ISOPAR-M, with the results shown in Fig. 15. The initial test sequence was performed at 150°C and is plotted in black. This temperature is likely to be slightly higher than that experienced by the unit injector in conventional diesel engines.<sup>(31)</sup> The remaining test conditions are detailed in TABLE 3.

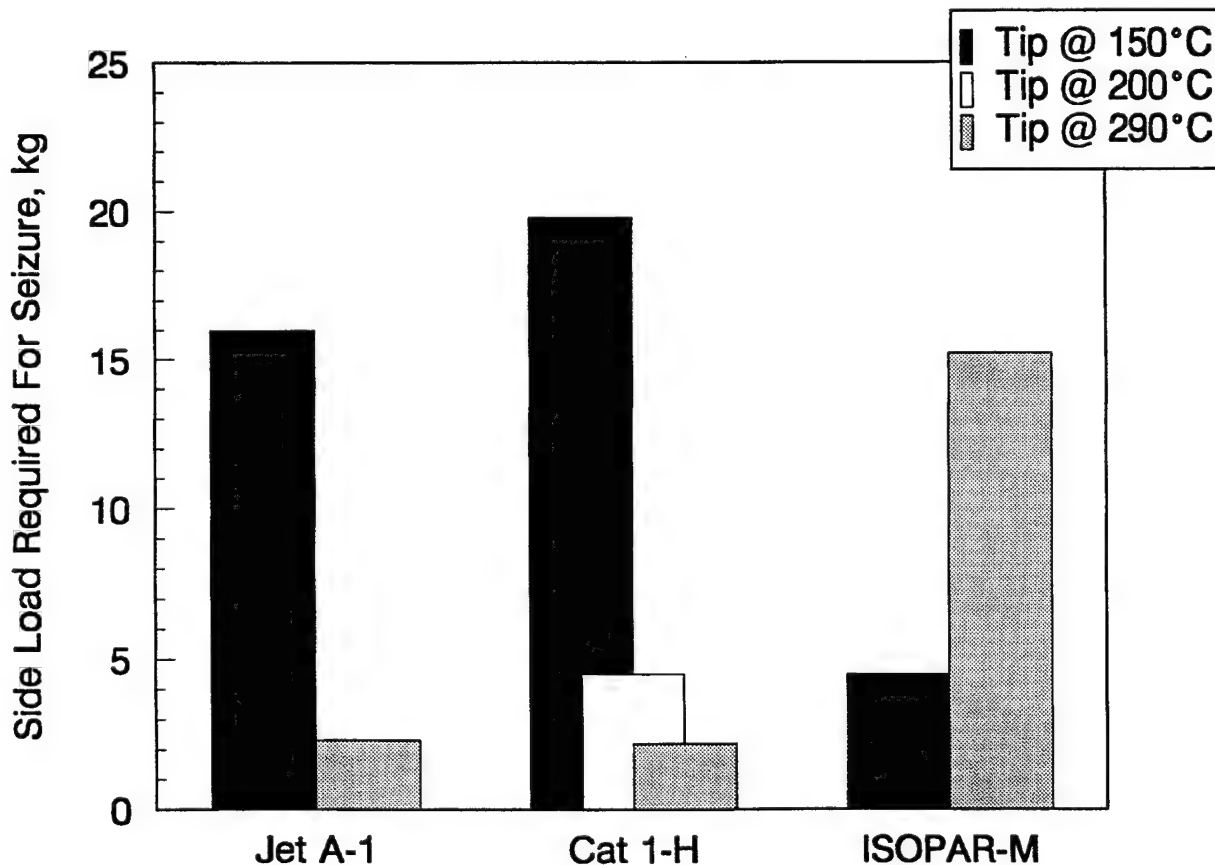


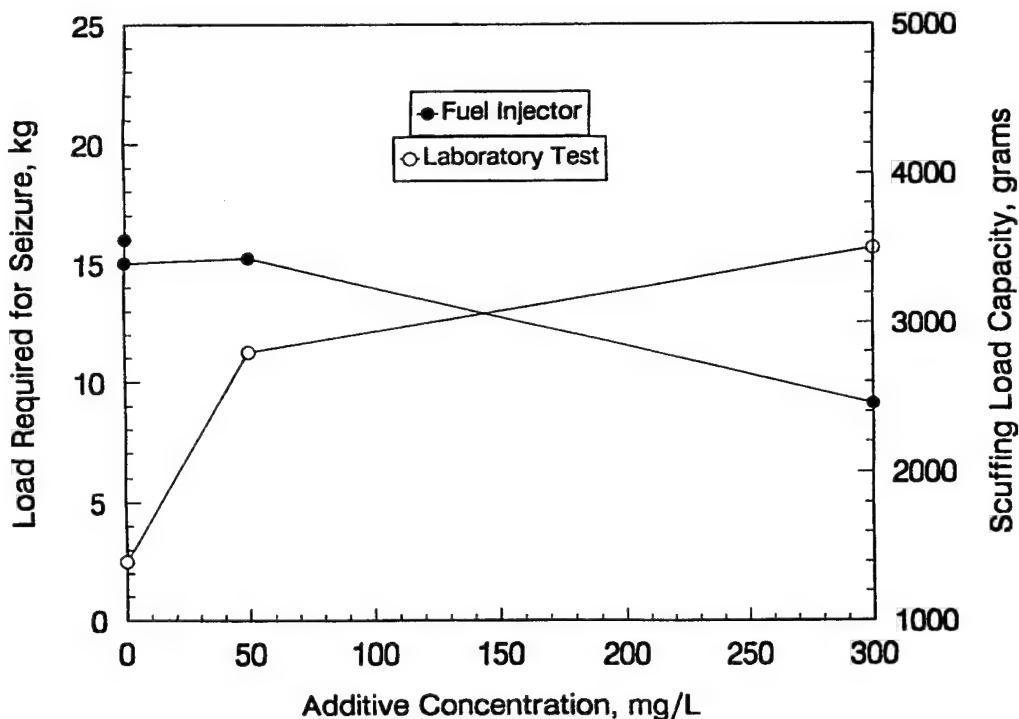
Figure 15. Side loading required for seizure with unadditized fuels

The Cat 1-H fuel provided the best performance of the group, probably due to its combined high viscosity and excellent lubricity. In contrast, failure occurred with Isopar M at a side loading of only 4 kg. Isopar M has very low lubricity but good viscosity. Jet A-1 required a relatively high side load of 16 kg to promote seizure. A repeat test performed with Jet A-1 under similar test conditions produced an identical result. The Jet A-1 fuel used has both low viscosity and poor boundary lubricating characteristics at room temperature. However, the laboratory-scale wear tests detailed in Figs. 13 and 14 indicate that the boundary lubricating characteristics of Jet A-1 are improved at high temperatures.

The second test sequence was performed at an injector tip temperature of 290°C. This temperature is appreciably higher than that present in current engines and may be similar to that produced in future applications. The side load required to produce failure was significantly reduced compared to the previous tests at 150°C in injectors operating on both Jet A-1 and Cat 1-H. An additional test performed at 200°C with Cat 1-H also showed a considerable decrease in seizure resistance. Conversely, the failure load of unit injectors operating on Isopar M was significantly increased, compared to the previous test performed at 150°C. No reason for the improved lubricity of this fluid may be suggested other than formation of chemically active or polar species during the high temperature test.

The test sequence shown in Fig. 15 was performed with three fuels of widely varying viscosity and boundary lubricating characteristics. As a result, the independent effects of chemical lubricity and viscosity may not be separated. Additional full-scale equipment tests were performed with Jet A-1 that contained various concentrations of DCI-4A lubricity additive. This additive affects the fuel's boundary lubricating characteristics without changing viscosity. These tests were performed at an injector tip temperature of 150°C.

The side load required for seizure of the injector is plotted in Fig. 16 as a function of additive concentration. Fuel lubricity, as measured using laboratory wear tests performed at room temperature, is significantly increased by additive concentrations of up to 300 PPM. Conversely, the side load required to produce seizure of the Detroit diesel unit injector at 150°C is largely



**Figure 16. Effect of lubricity additive concentration on the Army SLWT and the side load required to produce scuffing in the unit injector system**

unaffected by additive concentration. Indeed, the side loading required decreases at high additive concentration, possibly due to an anomaly in the test results. It is likely that the dilineoleic acid-based additive desorbs at high operating temperatures and has little effect on lubricity under these conditions. This is in good directional agreement with nonstandard, higher temperature laboratory-scale wear tests.

## V. DISCUSSION

The predominant source of lubricity in diesel fuels is minority constituents, which are most likely to interact with fuel-wetted surfaces. These constituents are suspected to be the more polar molecules that contain heteroatoms and tertiary carbon atoms. It is precisely these molecules that are eliminated when diesel stocks are hydrogenated to improve emissions performance. The identities of the minor constituents are not known but should vary as sulfur and aromatics concentrations are reduced. As a result, it is likely that sulfur and aromatics content simply reflect the level of refining severity present, rather than being a direct measure of lubricity.

Moreover, the results of the present study indicate that the lubricity of a given fuel varies with increasing temperature.

The present report describes a database obtained using the previously validated Army SLWT procedure. A correlation between the Army SLWT and the HFRR is provided in Appendix A to allow more general application of the results. A nonlinear correlation is observed, indicating differing wear mechanisms in each instance. This database includes approximately 1,200 commercially available and experimental fuels and fuel additive combinations. As expected, almost all high-sulfur fuels evaluated were at or above the minimum lubricity requirement indicated by correlation with full-scale equipment in previous studies. However, many of the fuels barely exceeded the 3,000-gram minimum scuffing load capacity. Therefore, it may be assumed that empirical manufacturing experience has optimized the design criteria necessary to provide acceptable durability and performance while operating with these fuel types. As a result, any significant variations in fuel composition, such as a switch to arctic fuel or jet kerosene, have historically required a metallurgical or design modification.

The results contained in the database confirm that fuel lubricity is directionally correlated to aromatic and sulfur content, resulting in a general decrease in lubricity as more stringent emissions regulations are introduced. In some instances, this change in fuel composition is sufficient to reduce lubricity below the level recommended by the vehicle hardware manufacturers for acceptable durability. Indeed, a few low lubricity fuels, similar to those which originally caused durability problems in Sweden, are commercially available in the United States.<sup>1</sup> However, the random distribution of these fuels precludes retrofitting vehicle fuel injection systems with improved wear resistant metallurgies.

The majority of the laboratory-scale wear tests reported in the present study were performed at 25°C. Clearly, this temperature is lower than that seen in practical operation and is significantly below the operating temperature in future low heat rejection engines. Both laboratory-scale and

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<sup>1</sup> Early samples of very low sulfur Swedish fuel that did not contain lubricity additives produced a scuffing load capacity of 1,200 and 1,400 grams for the Class I and II grades, respectively. Later samples that contained lubricity additives produced an Army SLWT capacity in excess of 3,000 grams and did not cause durability problems.



full-scale equipment tests clearly indicated that fuel lubricity is affected by operating temperature and thermal prestressing. The effects of temperature appear to depend on fuel chemistry and probably involves desorption of boundary additives and formation of new surface-active compounds. However, high temperature laboratory-scale wear tests for fuels typically have poor repeatability, due in part to evaporation of lighter fuel fractions. Additional research is required to better define the effects of temperature on fuel-lubricated wear and also the correlation between laboratory-scale wear tests and full-scale equipment.

Fortunately, widespread catastrophic failures are not yet being observed in the United States. In contrast to Sweden, the average lubricity of diesel fuel in the current United States fuel supply is relatively high, with an average scuffing load capacity of approximately 3,200 grams (Jet A-1 and JP-8 kerosene fuels are likely to have appreciably lower lubricity). It is likely that inadvertent mixing of good and poor lubricity diesel fuels occurs in most commercial applications, resulting in a relatively good lubricity blend. Larger consumers, such as military bases (which depend on a single fuel source), are more vulnerable to the effects of the isolated, low lubricity diesel fuels on the market. As a result, such users are occasionally experiencing decreased durability from the introduction of low sulfur, low aromatic fuels.

## VI. CONCLUSIONS

- a) Very low lubricity was observed for a number of commercially available fuels meeting 1993 EPA regulations (i.e.,  $<0.05$  mass% sulfur). A number of the lowest lubricity fuels were independently reported to have reduced the durability of the fuel injection system on military vehicles. No field data are available for the remaining fuels.
- b) Some correlation was observed between the Army SLWT and the HFRR.
- c) The Army SLWT--and by inference, the HFRR test--provides a more severe pass/fail criteria than the procedure defined in ASTM D 5001, which is only sensitive to the lubricity of the most severely refined fuels. However, the two are not mutually contradictory.

- d) The great majority of high-sulfur fuels (i.e., >0.15 mass%) had good lubricity.
- e) Low-sulfur fuels (i.e., <0.05 mass%) had widely varying lubricity, with some showing poor wear characteristics.
- f) No unadditized fuel with a total aromatics content below 10 percent showed good lubricity.
- g) Many post-1993 fuels contained appreciably less than the 0.05 mass% sulfur mandated by the EPA. California fuels were particularly low.
- h) The aromatics content of most California fuels was above 10 mass%, although the average value remained appreciably below that observed for the remainder of the country.
- i) Lubricity additives are effective in improving scuffing load capacity at concentrations above 75 to 100 mg/L and have the greatest effect in low lubricity fuels.
- j) Good directional correlation was observed between fuel lubricity (measured as scuffing load capacity) and diaromatic and polyaromatic concentrations for fuels that do not contain additives.
- k) A slight directional correlation was observed between viscosity and scuffing load capacity.
- l) The effects of most lubricity additives evaluated are not significantly better than those produced by di-carboxylic acid compounds, such as di-linoleic acid.
- m) Poor lubricity fuels may be improved through blending with a small amount of better lubricity fuel, although the effects appear fuel composition-sensitive. Conversely, blending of low lubricity kerosene fuels, as is common in arctic conditions, will reduce the lubricity of the base fuel.

- n) Biodiesel fuels consisting of methyl esters of soybean oil had excellent scuffing and adhesive wear resistance that exceeds those of the best conventional diesel fuels.
- o) Average lubricity of fuels from the major California refineries is almost identical to the lubricity of fuels found in the remainder of the United States, probably due to extensive use of additives.
- p) Some very low lubricity fuels are commercially available. However, their effect is probably masked by in-vehicle blending with better lubricity fuels.
- q) Operating temperature has a significant effect on lubricity. However, repeatability of laboratory-scale wear tests is often reduced at higher temperatures.
- r) The boundary lubricating characteristics of Jet A-1 improve at higher operating temperatures.
- s) Lubricity additives such as those in JP-8, which are effective at room temperature, are less effective under high temperature operating conditions.
- t) No direct correlation was observed between the high temperature seizure resistance of unit fuel injectors and laboratory wear tests performed at room temperature.
- u) No direct correlation was observed between the high temperature seizure resistance of unit fuel injectors and viscosity measured at 40°C.
- v) Thermal prestressing of fuels using a single tube heat exchanger significantly reduced oxidative corrosive wear of low lubricity Jet A-1 that predominates under lightly loaded conditions such as in the ASTM D 5001 test. A slight increase in wear rate was observed with some high-sulfur fuels under these conditions, probably due to formation of reactive compounds.

## VII. RECOMMENDATIONS

- a) Develop a laboratory wear test apparatus that allows accurate high-temperature wear testing and eliminates evaporation of light fractions from the fuel.
- b) Define the effects of increasing temperature on the lubricity of different fuels (i.e., Jet A-1, a low-sulfur diesel, and a high-sulfur diesel) using laboratory wear tests.
- c) Define the effects of increasing temperature on the effectiveness of commercially available lubricity additives using laboratory-scale wear tests. Particular attention should be given to additives qualified under MIL-I-25017.
- d) Perform full-scale equipment tests as a function of temperature. Particular attention should be given to those fuels previously shown to be sensitive to temperature using laboratory-scale wear tests. The effects of viscosity should also be defined.
- e) Define the naturally occurring compounds in the fuel that are responsible for lubricity. Such a study would require knowledge of the lubrication mechanisms present, as well as chemical analysis of the wear scars, fuels, and surface deposits.
- f) Define the effects of high molecular weight surface deposits generated during the wear process on lubricity. In particular, determine if these degradation products relate to fuel stability.
- g) Verify the correlation between laboratory-scale wear tests performed with lubricity additives and their effectiveness when used in full-scale equipment.

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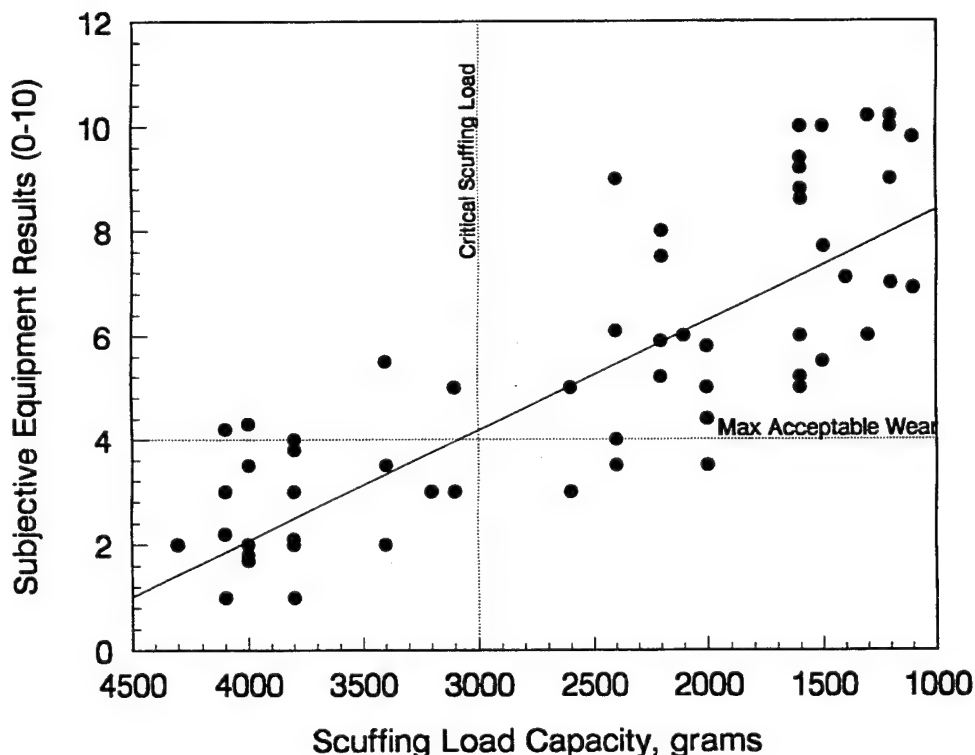
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**APPENDIX A**  
**Significance of Test Results**

## Significance of Test Results

The primary objective of the Army SLWT is to predict the effect of various fuels on equipment durability. Figure A-1 compares the Army SLWT results to wear produced in full-scale tests with four different equipment types, using identical fuels.(23, 35) Good correlation was observed, with an overall coefficient of determination of 0.66. Significantly higher correlations were observed among individual equipment types.(35) Moreover, the accuracy of the correlation is likely to be partially degraded by the accuracy of the equipment tests, which were performed at several locations, with widely different equipment, operating conditions, rating procedures, and lack of control of fuel moisture content. Some of the full-scale equipment tests were performed by the original manufacturers, who indicate that a subjective pump rating in excess of 4 corresponds to unacceptable field wear. This value corresponds to a result of approximately 3,000 grams in the Army SLWT.



(Note: Data includes neat and additized fuels in four equipment types.)

**Figure A-1. Relationship between full-scale fuel injection system wear and the Army SLWT with identical fuels**

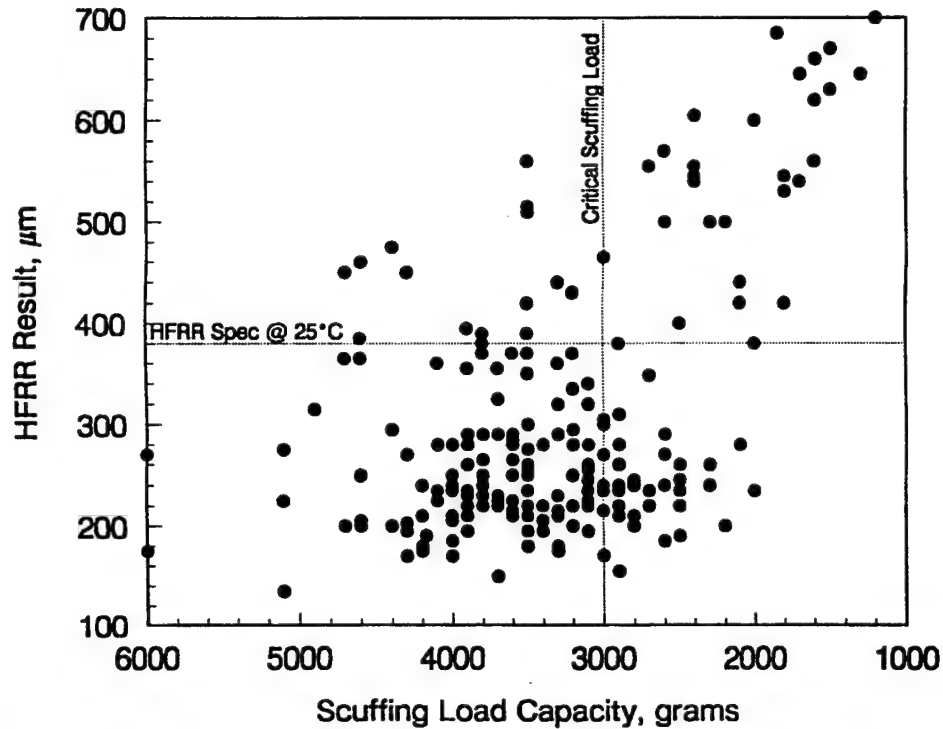
Clearly, a minimum applied load in excess of 3,000 grams indicates that on average, a fuel is likely to produce acceptable wear in full-scale equipment. However, this value is unlikely to be absolute and will vary as a function of fuel viscosity, operating temperature, and individual equipment requirements. As a result, a number of organizations have adopted slightly different minimum fuel lubricity requirements, based on the application and the margin of safety desired. The Army SLWT has recently been incorporated into the proposed revision E of Federal Specification VV-F-800 for diesel fuels (36) and is now used as an interim measurement of fuel lubricity by a number of fuel and engine manufacturers. (Note: Revision E has been withdrawn due to nonconcurrence by the Defense Fuel Supply Center, who is the Lead Service Activity for FSSC 9130/9140.) In addition, the test has been recommended as an interim minimum lubricity requirement for the state of California, pending acceptance of a final standard.(37)

The results obtained using the Army SLWT show some correlation with other recently developed tests intended to measure lubricity of diesel fuels. Correlation with the HFRR is shown in Fig. A-2. Correlation is observed with the HFRR test for low lubricity fuels, that produce a scuffing load capacity below 3,000 grams. Reduced correlation is observed at higher scuffing load capacities, partially due to the reduced ability of the HFRR to differentiate between these fuels. Significantly, the HFRR's minimum acceptable fuel lubricity limits of  $0.38 \text{ mm}^2$  (35) approximately correspond to the critical Army SLWT result of 3,000 grams used in the present report.

When the Army SLWT is compared to wear tests originally designed to measure lubricity of more severely refined fuels such as aviation kerosene, the degree of correlation is further reduced. For example, a nonlinear relationship exists between Army SLWT results and the ASTM D 5001 (24) test procedure, as shown in Fig. A-3. The ASTM D 5001 procedure is primarily directed toward an oxidative corrosion wear mechanism present only in the most severely refined fuels.(21) The oxidative mechanism is eliminated by trace quantities of natural or artificial compounds that act as corrosion inhibitors. In fact, this test is primarily used to qualify the very

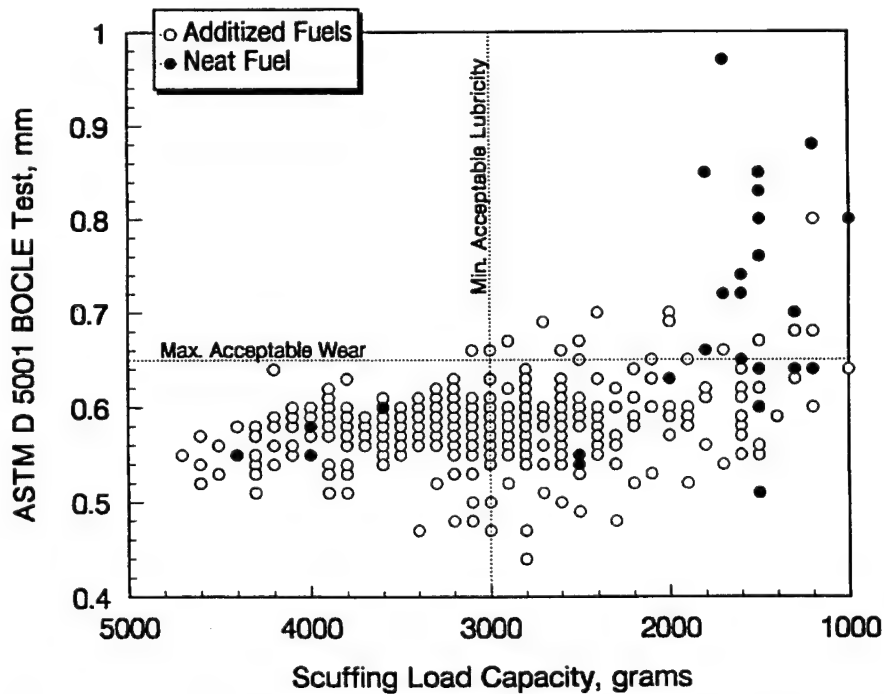
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<sup>2</sup> The HFRR test may be performed at either 25°C or 60°C. The HFRR data reported in the present study was obtained at 25°C.



(Note: The HFRR test may be performed at either 25°C or 60°C, as defined in TABLE 2.)

**Figure A-2. Correlation between the Army SLWT and the HFRR**



**Figure A-3. Correlation between the Army SLWT and results obtained in tests performed according to the ASTM D 5001 test procedure for the BOCLE**

low concentrations of corrosion inhibiting/lubricity additives specified in MIL-I-25017.(38) Further increases in additive concentration have no effect on the very mild ASTM D 5001 BOCLE test.

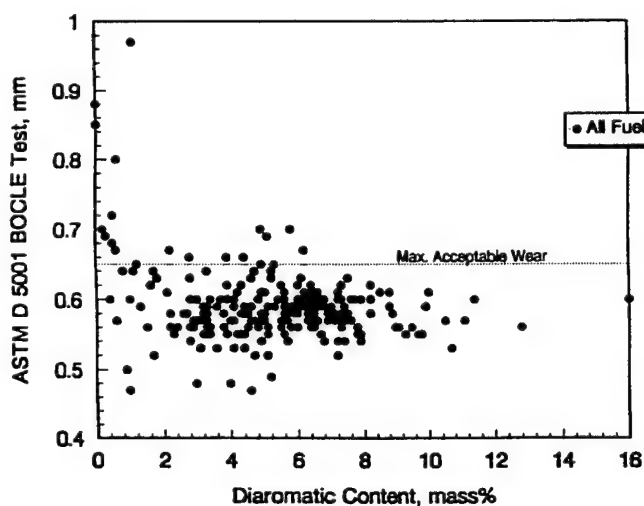
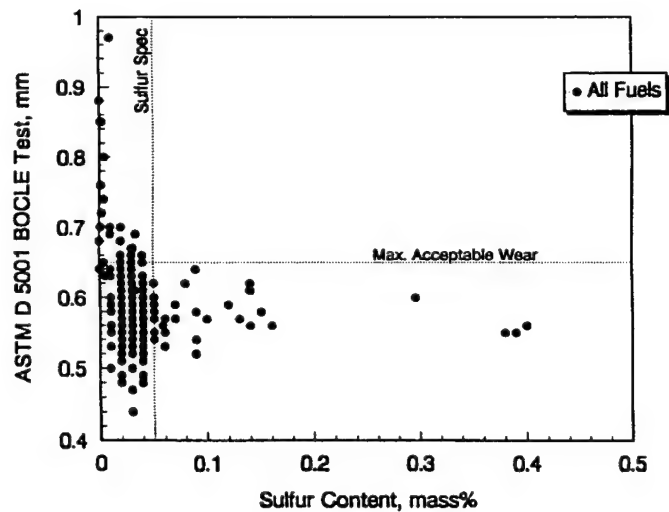
In reality, the load carrying ability of the fuel is improved by higher concentrations of anti-wear compounds, a fact reflected by the Army SLWT and HFRR test. As a result, the ASTM D 5001 BOCLE test is only capable of predicting the very poorest lubricity fuels, while the Army SLWT and HFRR remain viable over the complete spectrum of fuels. Indeed, consideration of Fig. A-3 indicates that only a very small percentage of fuels in the present matrix produced a wear scar greater than the critical level of 0.65 mm, specified in MIL-I-25017. In contrast, many more fuels failed to achieve the 3,000-gram minimum required for the Army SLWT, i.e., the lower right-hand quadrant of Fig. A-3. The two tests are not contradictory, and the ASTM D 5001 procedure only failed one fuel which was found to be acceptable by the Army SLWT, i.e., the upper left-hand quadrant of Fig. A-3.

Data obtained using the ASTM D 5001 procedure is plotted in Figs. A-4a, A-4b, and A-4c, as a function of sulfur, diaromatic, and polyaromatic concentration, respectively. (Note: Some fuels may contain additives.) Clearly, wear due to the oxidative corrosion mechanism is independent of fuel composition for all but the most severely refined fuels that contain very small concentrations of sulfur and aromatics compounds. Only the most severely refined fluids produce severe wear. A number of fuels with higher concentrations of diaromatic and polyaromatic compounds slightly exceeded the 0.65-mm wear limit, specified in MIL-I-25017.(38) However, none exceeded the limit by more than 0.05 mm, which approximates the test repeatability<sup>3</sup>.(24) The extreme sensitivity of the ASTM D 5001 test to trace concentrations of naturally occurring compounds mirrors the results obtained with artificial lubricity additives. These results appear to correlate well with the requirements of aviation equipment, which are designed to operate on highly processed kerosene fuels. Ground vehicles have been designed to operate on better lubricity diesel fuels and require a correspondingly more severe test.

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<sup>3</sup> It should also be noted that very high sulfur fuels occasionally exceed the 0.65 mm limit, due to sulfur corrosion which is distinct from oxidative corrosion. These fuels typically show good scuffing resistance.

a. Sulfur content



b. Diaromatic hydrocarbon content

c. Polyaromatic hydrocarbon content

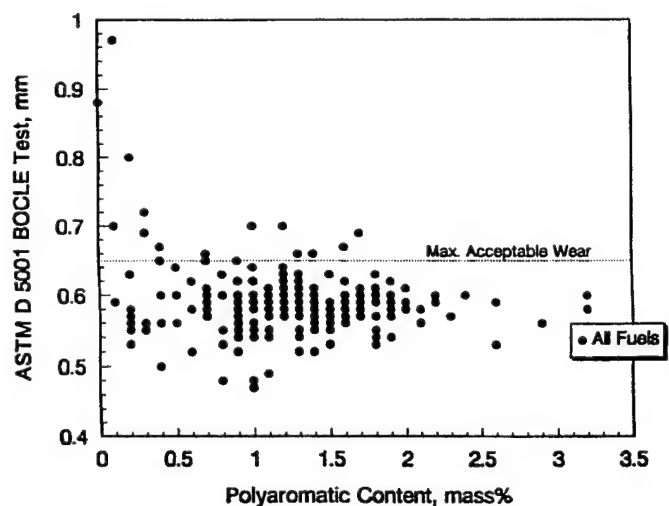


Figure A-4. Effect of fuel composition on wear measured using ASTM D 5001

**APPENDIX B**  
**Unit Injector Test System**

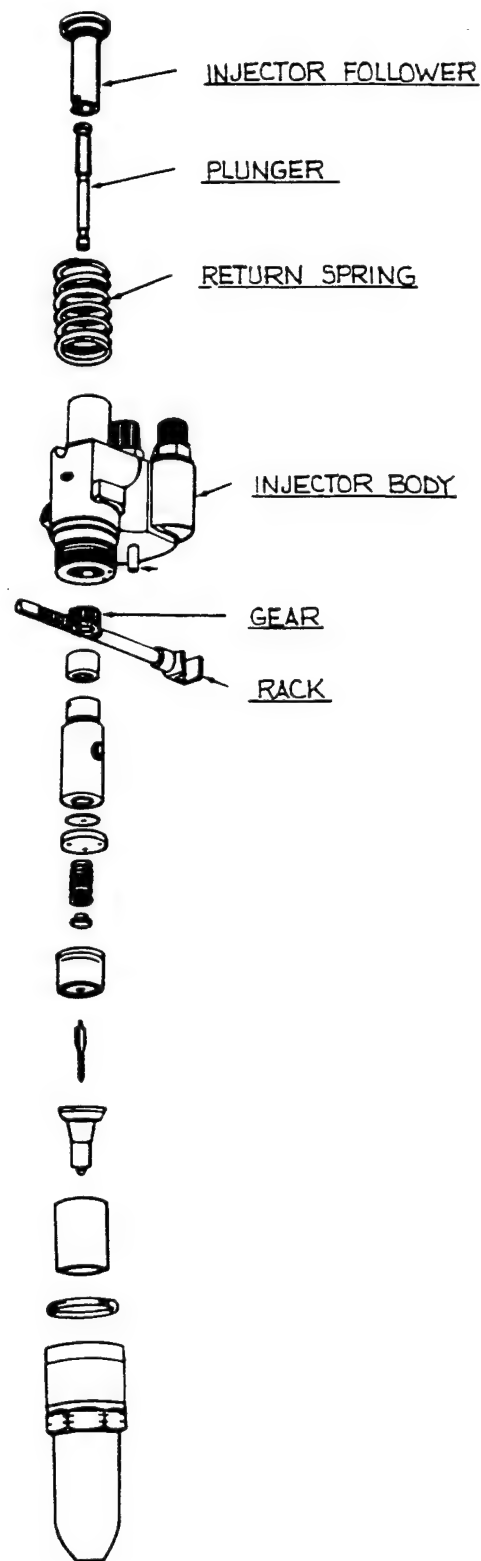
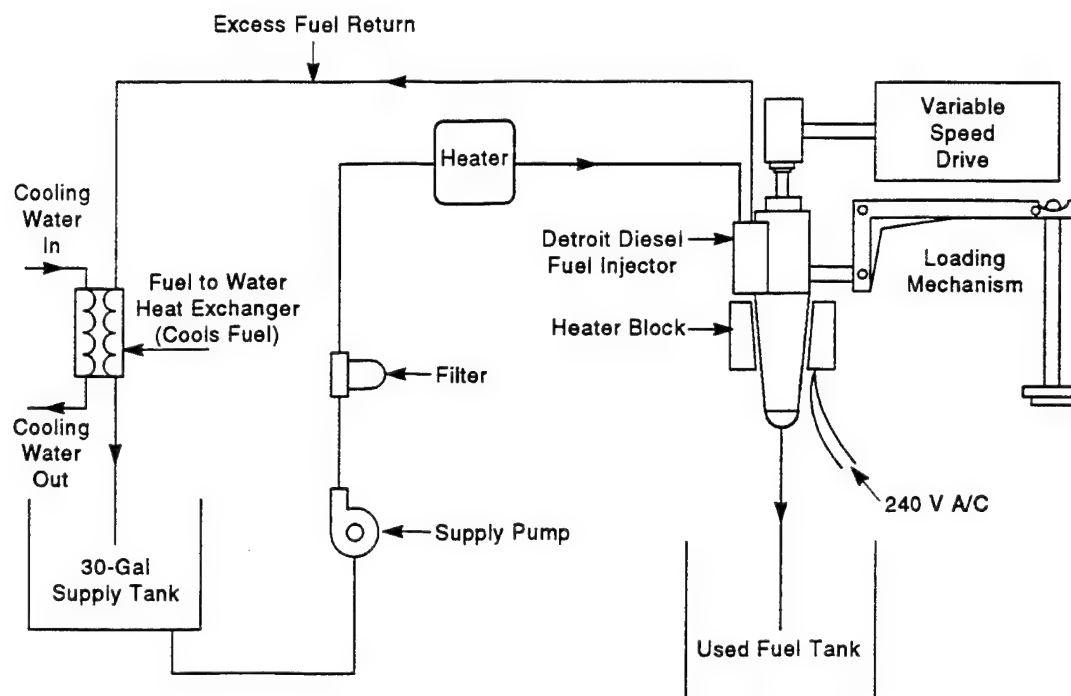


Figure B-1. Schematic diagram of the Detroit Diesel unit injector





**Figure B-2. Schematic diagram of the unit injector test loop**

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